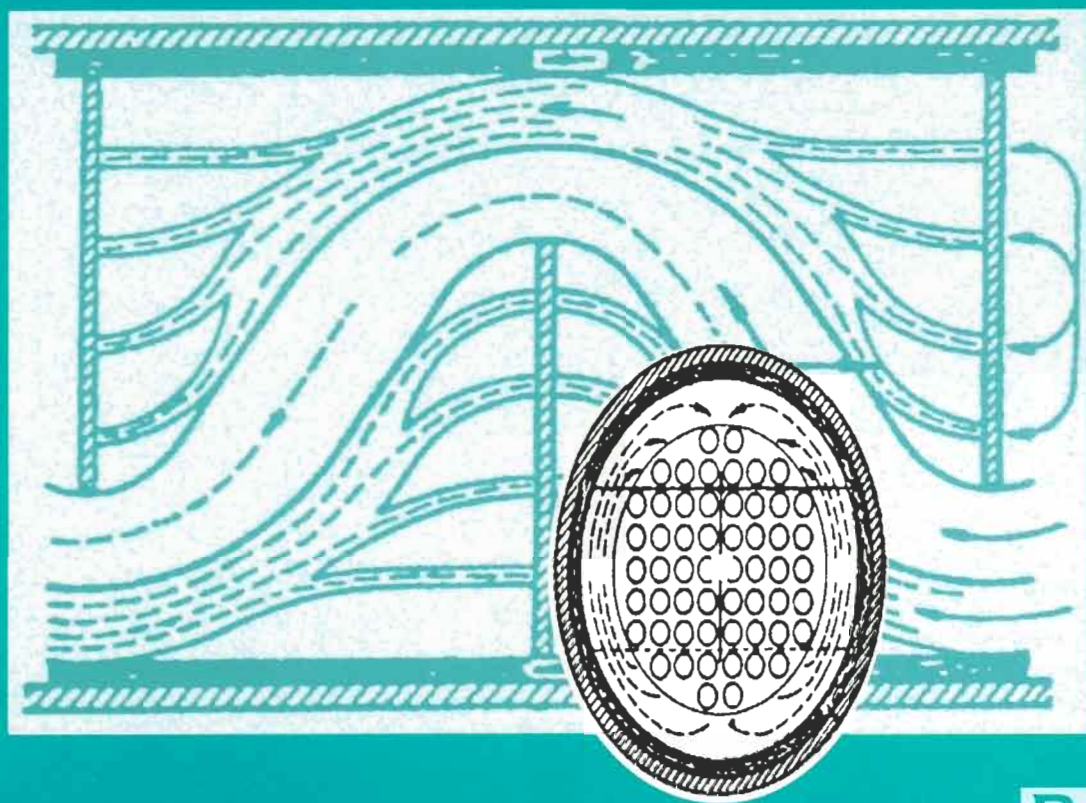# Handbook of Chemical Processing Equipment

Nicholas P. Cheremisinoff

Handbook of Chemical Processing Equipment

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CONTENTS

PREFACE

The chemical industry represents a 455-billion-dollar-a-year business, with products ranging from cosmetics, to fuel products, to plastics, to pharmaceuticals, health care products, food additives, and many others. It is diverse and dynamic, with market sectors rapidly expanding, and in turmoil in many parts of the world. Across these varied industry sectors, basic unit operations and equipment are applied on a daily basis, and indeed although there have been major technological innovations to processes, many pieces of equipment are based upon a foundation of engineering principles developed more than 50 years ago.

The *Handbook of Chemical Processing Equipment* has been written as a basic reference for process engineers. It provides practical information on the working principles and engineering basis for major equipment commonly used throughout the chemical processing and allied industries. Although written largely with the chemical engineer in mind, the book's contents are general enough, with sufficient background and principles described, that other manufacturing and process engineers will find it useful.

The handbook is organized into eight chapters. Chapters 1 through 3 deal with heat transfer equipment used in a variety of industry applications ranging from process heat exchange, to evaporative cooling, to drying and solvent recovery applications, humidity control, crystallization, and others. Chapters 4 and 5 cover stagewise mass transfer equipment. Specifically, Chapter 4 covers distillation, and Chapter 5 covers classical mass transfer equipment involving absorption, adsorption, extraction, and membrane technologies. Chapter 6 discusses equipment used in mass separation based upon physical or mechanical means. This includes such equipment topics as sedimentation, centrifugal separation, filtrations methods. Chapter 7 covers mixing equipment and various continuous contacting devices such as gas-solids fluidized beds. Finally, Chapter 8 provides the reader with a compendium of short calculation methods for commonly encountered process operations. The calculation methods are readily set up on a personal computer's standard software spreadsheet.

Select references are provided in each chapter for more in-depth coverage of an equipment subject, including key Web sites that offer vendor-specific information and equipment selection criteria. In a number of chapters, sample calculations are provided to guide the reader through the use of design and scale-up formulas that are useful in preparing equipment specifications or in establishing preliminary designs.

Although the author has taken great care to ensure that the information presented in this volume is accurate, neither he nor the publisher will endorse or guarantee any designs based upon materials provided herein. The author wishes to thank Butterworth-Heinemann Publishers for their fine production of this volume.

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Nicholas P. Cheremisinoff is a consultant to a number of organizations and private companies. Among his clients are the World Bank Organization, the International Finance Corporation, the United States Agency for International Development, Chemonics International, Booz-Allen & Hamilton, Inc., and several private sector clients. He has extensive business development, project financing, and engineering experience working in countries that were former Soviet Union republics, and has assisted in privatization and retooling industry with emphasis on environmentally sound practices. Although a chemical engineer by profession, his engineering and consulting experiences have spanned several industry sectors, including automotive manufacturing, mining, gas processing, plastics, and petroleum refining. He is a recognized authority on pollution prevention practices, and has led programs dealing with pollution prevention auditing, training in environmental management practices, development of environmental management plans, as well as technology and feasibility studies for environmental project financing through international lending institutions. He has contributed extensively to the industrial press, having authored, co-authored, or edited more than 100 technical books. Dr. Cheremisinoff received his B.S., M.S., and Ph.D. degrees from Clarkson College of Technology.

Chapter 1
HEAT EXCHANGE EQUIPMENT

INTRODUCTION

Prior to the 19th century, it was believed that the sense of how hot or cold an object felt was determined by how much "heat" it contained. Heat was envisioned as a liquid that flowed from a hotter to a colder object; this weightless fluid was called "caloric", and until the writings of Joseph Black (1728-1799), no distinction was made between heat and temperature. Black distinguished between the quantity (caloric) and the intensity (temperature) of heat. Benjamin Thomson, Count Rumford, published a paper in 1798 entitled "An Inquiry Concerning the Source of Heat which is Excited by Friction". Rumford had noticed the large amount of heat generated when a cannon was drilled. He doubted that a material substance was flowing into the cannon and concluded "it appears to me to be extremely difficult if not impossible to form any distinct idea of anything capable of being excited and communicated in the manner the heat was excited and communicated in these experiments except motion."

But it was not until J. P. Joule published a definitive paper in 1847 that the caloric idea was abandoned. Joule conclusively showed that heat was a form of energy. As a result of the experiments of Rumford, Joule, and others, it was demonstrated (explicitly stated by Helmholtz in 1847), that the various forms of energy can be transformed one into another.

When heat is transformed into any other form of energy, or when other forms of energy are transformed into heat, the total amount of energy (heat plus other forms) in the system is constant. This is known as the first law of thermodynamics, i.e., the conservation of energy. To express it another way: it is in no way possible either by mechanical, thermal, chemical, or other means, to obtain a perpetual motion machine; i.e., one that creates its own energy.

A second statement may also be made about how machines operate. A steam engine uses a source of heat to produce work. Is it possible to completely convert the heat energy into work, making it a 100% efficient machine? The answer is to be found in the second law of thermodynamics: No cyclic machine can convert heat energy wholly into other forms of energy. It is not possible to construct a cyclic machine that does nothing, but withdraw heat energy and convert it into mechanical energy. The second law of thermodynamics implies the irreversibility

of certain processes - that of converting all heat into mechanical energy, although it is possible to have a cyclic machine that does nothing but convert mechanical energy into heat.

Sadi Carnot (1796-1832) conducted theoretical studies of the efficiencies of heat engines (a machine which converts some of its heat into useful work). He was trying to model the most efficient heat engine possible. His theoretical work provided the basis for practical improvements in the steam engine and also laid the foundations of thermodynamics. He described an ideal engine, called the Carnot engine, that is the most efficient way an engine can be constructed. He showed that the efficiency of such an engine is given by:

$$\text{efficiency} = 1 - T''/T'$$

where the temperatures, T' and T'', are the cold and hot "reservoirs", respectively, between which the machine operates. On this temperature scale, a heat engine whose coldest reservoir is zero degrees would operate with 100% efficiency. This is one definition of absolute zero. The temperature scale is called the absolute, the thermodynamic , or the kelvin scale.

The way, that the gas temperature scale and the thermodynamic temperature scale are shown to be identical, is based on the microscopic interpretation of temperature, which postulates that the macroscopic measurable quantity called temperature, is a result of the random motions of the microscopic particles that make up a system.

About the same time that thermodynamics was evolving, James Clerk Maxwell (1831-1879) and Ludwig Boltzmann (1844-1906) developed a theory, describing the way molecules moved - molecular dynamics. The molecules that make up a perfect gas move about, colliding with each other like billiard balls and bouncing off the surface of the container holding the gas. The energy, associated with motion, is called Kinetic Energy and this kinetic approach to the behavior of ideal gases led to an interpretation of the concept of temperature on a microscopic scale.

The amount of kinetic energy each molecule has is a function of its velocity; for the large number of molecules in a gas (even at low pressure), there should be a range of velocities at any instant of time. The magnitude of the velocities of the various particles should vary greatly; no two particles should be expected to have the exact same velocity. Some may be moving very fast; others - quite slowly.

Maxwell found that he could represent the distribution of velocities statistically by a function, known as the Maxwellian distribution. The collisions of the molecules with their container gives rise to the pressure of the gas. By considering the average force exerted by the molecular collisions on the wall, Boltzmann was able to show that the average kinetic energy of the molecules was

directly comparable to the measured pressure, and the greater the average kinetic energy, the greater the pressure.

From Boyles' Law, it is known that the pressure is directly proportional to the temperature, therefore, it was shown that the kinetic energy of the molecules related directly to the temperature of the gas. A simple thermodynamic relation holds for this:

$$\text{average kinetic energy of molecules} = 3kT/2$$

where k is the Boltzmann constant. Temperature is a measure of the energy of thermal motion and, at a temperature of zero, the energy reaches a minimum (quantum mechanically, the zero-point motion remains at $0\ ^\circ K$).

About 1902, J. W. Gibbs (1839-1903) introduced statistical mechanics with which he demonstrated how average values of the properties of a system could be predicted from an analysis of the most probable values of these properties found from a large number of identical systems (called an ensemble). Again, in the statistical mechanical interpretation of thermodynamics, the key parameter is identified with a temperature, which can be directly linked to the thermodynamic temperature, with the temperature of Maxwell's distribution, and with the perfect gas law.

Temperature becomes a quantity definable either in terms of macroscopic thermodynamic quantities, such as heat and work, or, with equal validity and identical results, in terms of a quantity, which characterized the energy distribution among the particles in a system. With this understanding of the concept of temperature, it is possible to explain how heat (thermal energy) flows from one body to another.

Thermal energy is carried by the molecules in the form of their motions and some of it, through molecular collisions, is transferred to molecules of a second object, when put in contact with it. This mechanism for transferring thermal energy is called conduction.

A second mechanism of heat transport is illustrated by a pot of water set to boil on a stove - hotter water closest to the flame will rise to mix with cooler water near the top of the pot. Convection involves the bodily movement of the more energetic molecules in a liquid or gas. The third way, that heat energy can be transferred from one body to another, is by radiation; this is the way that the sun warms the earth. The radiation flows from the sun to the earth, where some of it is absorbed, heating the surface.

These historical and fundamental concepts form the foundation for the design, applications, and operations of a major class of equipment that are used throughout the chemical process industries - heat exchange equipment, or heat exchangers. There are many variations of these equipment and a multitude of

applications. However, the design configurations for these equipment are universal, meaning that they generally are not specific to a particular industry sector. In the United States in 1998, the chemical process industries (CPI) invested more than $700 million in capital equipment related to heat transfer. Much of that investment was driven by a growing body of environmental legislation, such as the U.S. Clean Air Act Amendments. The use of vent condensers, for example, which use heat exchangers to reduce the volume of stack emissions, is increasing. Heat exchanger makers have responded to growing environmental concerns over fugitive emissions, as well by developing a new breed of leak-tight heat exchangers, designed to keep process fluids from leaking and volatile organic compounds from escaping to the atmosphere. Gasketed exchangers are benefitting from improvements in the quality and diversity of elastomer materials and gasket designs. The use of exchangers with welded connections, rather than gaskets, is also reducing the likelihood of process fluid escape. Throughout the 1990's, the use of heat exchangers has expanded into non-traditional applications. This, coupled with a variety of design innovations, has given chemical engineers a wider variety of heat exchanger options to choose from than ever before. Operating conditions, ease of access for inspection and maintenance, and compatibility with process fluids are just some of the variables CPI engineers must consider when assessing heat exchanger options. Other factors include: maximum design pressure and temperature, heating or cooling applications, maintenance requirements, material compatibility with process fluids, gasket compatibility with process fluids, cleanliness of the streams, and temperature approach. This chapter provides an overview of the most commonly employed equipment. Emphasis is given to practical features of these systems, and typical examples of industrial applications are discussed.

GENERAL CONCEPTS OF HEAT TRANSFER

Before discussing typical equipment commonly used throughout the chemical processing industries, some general concepts and definitions regarding the subject of heat transfer are reviewed. The term **heat** in physics, refers to the transfer of energy from one part of a substance to another, or from one object to another, because of a difference in temperature. Heat flows from a substance at a higher temperature to a substance at a lower temperature, provided the volume of the objects remains constant. Heat does not flow from a lower to a higher temperature, unless another form of energy transfer, work, is also present.

Until the beginning of the 19th century, it was thought that heat was an invisible substance called caloric. An object at a high temperature was thought to contain more caloric than one at a low temperature. However, British physicist Benjamin Thompson in 1798 and British chemist Sir Humphry Davy in 1799 presented

evidence that heat, like work, is a form of energy transfer. In a series of experiments between 1840 and 1849, British physicist James Prescott Joule provided conclusive evidence that heat is a form of energy in transit, and that it can cause the same changes as work.

The sensation of warmth or coldness is caused by temperature. Adding heat to a substance not only raises its temperature, but also produces changes in several other qualities. The substance expands or contracts; its electric resistance changes; and in the gaseous form, its pressure changes. Five different temperature scales are in use today: Celsius, Fahrenheit, Kelvin, Rankine, and international thermodynamic.

The term **resistance** refers to the property of any object or substance to resist or oppose the flow of an electrical current. The unit of resistance is the ohm. The abbreviation for electric resistance is R and the symbol for ohms is the Greek letter omega, Ω. For certain electrical calculations the reciprocal of resistance is used, $1/R$, which is termed conductance, G. The unit of conductance is the mho, or ohm spelled backward, and the symbol is an inverted omega.

Pressure, in mechanics, is the force per unit area exerted by a liquid or gas on an object or surface, with the force acting at right angles to the surface and equally in all directions. In the United States, pressure is usually measured in pounds per square inch (psi); in international usage, in kilograms per square centimeters, or in atmospheres; and in the international metric system (SI), in newtons per square meter (International System of Units). Most pressure gauges record the difference between a fluid pressure and local atmospheric pressure. Types of common pressure gauges include U-tube manometers, for measuring small pressure differences; Bourdon gauges, for measuring higher pressure differences; gauges that use piezoelectric or electrostatic sensing elements, for recording rapidly changing pressures; McLeod gauges, for measuring very low gas pressures; and gauges that use radiation, ionization, or molecular effects to measure low gas pressures (in vacuum technology). In the atmosphere the decreasing weight of the air column with altitude leads to a reduction in local atmospheric pressure. Partial pressure is the effective pressure that a single gas exerts in a mixture of gases. In the atmosphere the total pressure is equal to the sum of the partial pressures.

Heat is measured in terms of the calorie, defined as the amount of heat necessary to raise the temperature of 1 gram of water at a pressure of 1 atmosphere from 15° to 16 °C. This unit is sometimes called the small calorie, or gram calorie, to distinguish it from the large calorie, or kilocalorie, equal to 1000 small calories, which is used in nutritional studies. In mechanical engineering practice in the United States and the United Kingdom, heat is measured in British thermal units (Btu). One Btu is the quantity of heat required to raise the temperature of 1 pound of water 1° F and is equal to 252 calories.

The term **latent heat** is also pertinent to our discussions. The process of changing from solid to gas is referred to as sublimation; from solid to liquid, as melting; and from liquid to vapor, as vaporization. The amount of heat required to produce such a change of phase is called latent heat. If water is boiled in an open container at a pressure of 1 atmosphere, its temperature does not rise above 100° C (212° F), no matter how much heat is added. The heat that is absorbed without changing the temperature is latent heat; it is not lost, but is expended in changing the water to steam.

The **phase rule** is a mathematical expression that describes the behavior of chemical systems in equilibrium. A chemical system is any combination of chemical substances. The substances exist as gas, liquid, or solid phases. The phase rule applies only to systems, called heterogeneous systems, in which two or more distinct phases are in equilibrium. A system cannot contain more than one gas phase, but can contain any number of liquid and solid phases. An alloy of copper and nickel, for example, contains two solid phases. The rule makes possible the simple correlation of very large quantities of physical data and limited prediction of the behavior of chemical systems. It is used particularly in alloy preparation, in chemical engineering, and in geology.

The subject of heat transfer refers to the process by which energy in the form of heat is exchanged between objects, or parts of the same object, at different temperatures. Heat is generally transferred by radiation, convection, or conduction, processes that may occur simultaneously.

Conduction is the only method of heat transfer in opaque solids. If the temperature at one end of a metal rod is raised, heat travels to the colder end. The mechanism of conduction in solids is believed to be partially due to the motion of free electrons in the solid matter. This theory helps explain why good conductors of electricity also tend to be good conductors of heat. In 1882 French mathematician Jean Baptiste Joseph Fourier formulated a law that the rate, at which heat is conducted through an area of an object, is proportional to the negative of the temperature change through the object. Conduction also occurs between two objects, if they are brought into contact. Conduction between a solid surface and a moving liquid or gas is called convection. The motion of the fluid may be natural or forced. If a liquid or gas is heated, its mass per unit of volume generally decreases. If the substance is in a gravitational field, the hotter, lighter fluid rises while the colder, heavier fluid sinks. This kind of motion is called natural **convection**. Forced convection is achieved by putting the fluid between different pressures, and so forcing motion to occur according to the law of fluid mechanics.

Radiation is a process that is different from both conduction and convection, because the substances exchanging heat need not be touching and can even be separated by a vacuum. A law formulated by German physicist Max Planck in

1900 states, in part, that all substances emit radiant energy, simply because they have a positive absolute temperature. The higher the temperature, the greater the amount of energy emitted. In addition to emitting, all substances are capable of absorbing radiation. The absorbing, reflecting, and transmitting qualities of a substance depend upon the wavelength of the radiation.

In addition to heat transfer processes that result in raising or lowering temperatures, heat transfer can also produce phase changes in a substance, such as the melting of ice. In engineering, heat transfer processes are usually designed to take advantage of this ability. For instance, a space capsule reentering the atmosphere at very high speeds is provided with a heat shield that melts to prevent overheating of the capsule's interior. The frictional heat, produced by the atmosphere, is used to turn the shield from solid to liquid and does not raise the temperature of the capsule.

Evaporation is the gradual change of a liquid into a gas without boiling. The molecules of any liquid are constantly moving. The average molecular speed depends on the temperature, but individual molecules may be moving much faster or slower than the average. At temperatures below the boiling point, faster molecules approaching the liquid's surface may have enough energy to escape as gas molecules. Because only the faster molecules escape, the average speed of the remaining molecules decreases, lowering the liquid's temperature, which depends on the average speed of the molecules.

An additional topic to discuss from an introductory standpoint is **thermal insulating materials**. These materials are used to reduce the flow of heat between hot and cold regions. The sheathing often placed around steam and hot-water pipes, for instance, reduces heat loss to the surroundings, and insulation placed in the walls of a refrigerator reduces heat flow into the unit and permits it to stay cold.

Thermal insulation generally has to fulfill one or more of three functions: to reduce thermal conduction in the material where heat is transferred by molecular or electronic action; to reduce thermal convection currents, which can be set up in air or liquid spaces; and to reduce radiation heat transfer where thermal energy is transported by electromagnetic waves. Conduction and convection can be suppressed in a vacuum, where radiation becomes the only method of transferring heat. If the surfaces are made highly reflective, radiation can also be reduced. As examples, thin aluminum foil can be used in building walls, and reflecting metal on roofs minimizes the heating effect of the sun. Thermos bottles or Dewar flasks provide insulation through an evacuated double-wall arrangement in which the walls have reflective silver or aluminum coatings. Air offers resistance to heat flow at a rate about 15,000 times higher than that of a good thermal conductor, such as silver, and about 30 times higher than that of glass.

Typical insulating materials, therefore, are usually made of nonmetallic materials and are filled with small air pockets. They include magnesium carbonate, cork, felt, cotton batting, rock or glass wool, and diatomaceous earth. Asbestos was once widely used for insulation, but it has been found to be a health hazard and has, therefore, been banned in new construction in the U.S.

In building materials, air pockets provide additional insulation in hollow glass bricks, insulating or thermopane glass (two or three sealed glass panes with a thin air space between them), and partially hollow concrete tile. Insulating properties are reduced, if the air space becomes large enough to allow thermal convection, or, if moisture seeps in and acts as a conductor. The insulating property of dry clothing, for example, is the result of air entrapped between the fibers; this ability to insulate can be significantly reduced by moisture. Home-heating and air-conditioning costs can be reduced by proper building insulation. In cold climates about 8 cm (about 3 in.) of wall insulation and about 15 to 23 cm (about 6 to 9 in.) of ceiling insulation are recommended. The effective resistance to heat flow is conventionally expressed by its R-value (resistance value), which should be about 11 for wall and 19 to 31 for ceiling insulation.

Superinsulation has been developed, primarily for use in space, where protection is needed against external temperatures near absolute zero. Superinsulation fabric consists of multiple sheets of aluminized mylar, each about 0.005 cm (about 0.002 in.) thick, and separated by thin spacers with about 20 to 40 layers per cm (about 50 to 100 layers per in.).

Governing Expressions for Heat Exchangers

When a hot fluid stream and a cold fluid stream, separated by a conducting wall, exchange heat, the heat that is transferred across a differential element can be represented by the following expression (refer to Figure 1):

$$dq = U \, \Delta t \, dA$$

where dq = heat transferred across differential element dA (W),

U = Overall heat transfer coefficient (W/m-°K),

Δt = temperature difference across element dA (°K),

dA = heat transfer area for the differential element (m^2).

The expression can be integrated over the entire heat exchanger using the simplification that the changes in U with temperature and position are negligible.
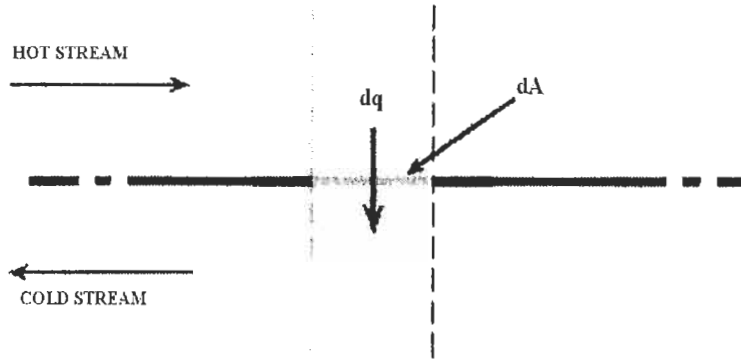
Figure 1. *Heat exchange across a differential element in a heat exchanger.*

In this manner, an average value of U can be applied to the whole exchanger. Ideally, the heat lost by the hot fluid stream is transferred totally to the cold stream, and hence, integrating results in the following expression:

$$q = UA\Delta t_{lm}$$

where A = the total heat exchange area (m^2), q = the total heat transferred (W), and U = the overall heat transfer coefficient, assumed to be constant throughout the exchanger (W/m^2-°K). The parameter Δt_{lm} is the log-mean temperature difference (in units of °K) and defined by the following expression:

$$\Delta t_{lm} = \Theta/\Gamma$$

where $\quad \Theta = (t_{h,in} - t_{c,out}) - (t_{h,out} - t_{c,in})$

$\quad\quad\quad \Gamma = \ln ((t_{h,in} - t_{c,out})/(t_{h,out} - t_{c,in}))$

The overall heat transfer coefficient, U, is a measure of the conductivity of all the materials between the hot and cold streams. For steady state heat transfer through the convective film on the outside of the exchanger pipe, across the pipe wall and through the convective film on the inside of the convective pipe, the overall heat transfer coefficient may be stated as:

$$1/U = A/h_1 A_1 + A\Delta x/k A_{lm} + A/h_2 A_2$$

where   A = a reference area (m^2),

h_1 = heat transfer coefficient inside the pipe (W/m²-°K),

A_1 = area inside the pipe (m^2),

Δx = pipe wall thickness (m),

k = thermal conductivity of the pipe (W/m-°K),

h_2 = heat transfer coefficient outside the pipe (W/m²-°K),

A_2 = area outside the pipe (m^2).

The term A_{lm} is the log-mean area of the pipe (in m^2) defined as follows:

$$A_{lm} = (A_1 - A_2)/\ln(A_1/A_2)$$

Estimation of the heat transfer coefficients for forced convection of a fluid in pipes is usually based on empirical expressions. The most well known expression for this purpose is:

$$Nu = 0.023 \, Re^{0.8} \, Pr^{0.33}$$

where Nu is the Nusselt number, a dimensionless group defining the relative significance of the film heat transfer coefficient to the conductivity of the pipe wall, Re is the Reynolds number, which relates inertial forces to viscous forces and thereby characterizes the type of flow regime, and Pr is the Prandtl number, which relates the thermal properties of the fluid to the conductivity of the pipe.

It is well known from heat transfer studies that the fluid heat transfer coefficient, h_1, is proportional to the velocity, v, of the fluid raised to the power 0.8. If all other parameters are kept constant, it then follows that a plot of $1/v^{0.8}$ versus $1/U$ results in a straight line with an intercept, representing the sum of the vapor film conductance and the wall conductance. Knowing the wall conductance, the vapor film conductance can be determined from the intercept value. Many of the properties used in the empirical expression are functions of temperature. In general, the properties needed to evaluate the above empirical expression are taken at the mean bulk temperature of the fluid, i.e., the average between the inlet and outlet temperatures. For water however, a temperature correction must be applied. The temperature corrected plot for water would be $1/(1+0.011t)v^{0.8}$ versus $1/U$, where t is the average fluid temperature measured in °F. The resulting plot should be linear for each separate steam pressure, thereby producing a series of lines with the same slope, but having a different intercept, that is a function of pressure.

Another area to consider is heat exchanger efficiency. The concept of efficiency is to compare the actual performance of a piece of equipment with the ideal performance (i.e., the maximum potential heat transfer). The maximum heat transfer possible is established by the stream that has the minimum heat capacity. That is the minimum value for the product of stream mass flowrate and specific heat. This stream would, for maximum heat transfer, leave the exchanger at the inlet temperature of the other stream. In terms of the hot stream, the efficiency can be stated as:

$$e = C_{p,h}m_h(t_{h,in} - t_{h,out})/((C_pm)_{min}(t_{h,in} - t_{c,in}))$$

And, in terms of the cold stream:

$$e = C_{p,c}m_c(t_{c,in} - t_{c,out})/((C_pm)_{min} (t_{h,in} - t_{c,in}))$$

In the above expressions:

e = heat exchanger efficiency,

$t_{h,in}$ = the inlet temperature of the hot stream (°K),

$t_{c,out}$ = the outlet temperature of the cold stream (°K),

$t_{h,out}$ = the outlet temperature of the hot stream (°K),

$t_{c,in}$ = the inlet temperature of the cold stream (°K),

$C_{p,h}m$ = the product of the hot stream heat capacity and the mass flowrate,

$C_{p,c}m$ = the product of the cold stream heat capacity and the mass flowrate,

$(C_pm)_{min}$ = the minimum product of stream heat capacity and mass flowrate.

Knowing the efficiency, one can use this value to predict heat exchanger performance for other streams and fluids. Efficiency is based on the maximum amount of heat that can be transferred:

$$q = e(C_pm)_{min} (t_{h,in} - t_{c,in})$$

AIR COOLED HEAT EXCHANGERS

Air cooled heat exchangers are used to transfer heat from a process fluid to ambient air. The process fluid is contained within heat conducting tubes. Atmospheric air, which serves as the coolant, is caused to flow perpendicularly across the tubes in order to remove heat. In a typical air cooled heat exchanger, the ambient air is either forced or induced by a fan or fans to flow vertically across a horizontal section of tubes. For condensing applications, the bundle may be sloped or vertical. Similarly, for relatively small air cooled heat exchangers, the air flow may be horizontal across vertical tube bundles.

In order to improve the heat transfer characteristics of air cooled exchangers, the tubes are provided with external fins. These fins can result in a substantial increase in heat transfer surface. Parameters such as bundle length, width and number of tube rows vary with the particular application as well as the particular finned tube design.

The choice of whether air cooled exchangers should be used is essentially a question of economics including first costs or capital costs, operating and maintenance expenses, space requirements, and environmental considerations; and involves a decision weighing the advantages and disadvantages of cooling with air.

The advantages of cooling with air may be seen by comparing air cooling with the alternative of cooling with water. The primary advantages and disadvantages of air cooled heat exchangers are summarized in Table 1. These issues should be examined on a case by case basis to assess whether air cooled systems are economical and practical for the intended application. Specific systems are described later in this chapter. The major components of air cooled heat exchangers include the finned tube, the tube bundle, the fan and drive assembly, an air plenum chamber, and the overall structural assembly. Each component is briefly described below.

Finned Tubes

Common to all air cooled heat exchangers is the tube, through which the process fluid flows. To compensate for the poor heat transfer properties of air, which flows across the outside of the tube, and to reduce the overall dimensions of the heat exchanger, external fins are added to the outside of the tube. A wide variety of finned tube types are available for use in air cooled exchangers. These vary in geometry, materials, and methods of construction, which affect both air side thermal performance and air side pressure drop. In addition, particular

combinations of materials and/or fin bonding methods may determine maximum design temperature limitations for the tube and limit environments, in which the tube might be used. The use of a particular fin tube is essentially a matter of agreement between the air cooled heat exchanger manufacturer and the user. Finned tubes may differ in the means, by which the fins themselves are attached or bonded to the bare tube.

Table 1. *Advantages and Disadvantages of Air Cooled Heat Exchange Devices*

Advantages
Since water is not used as the cooling medium, the disadvantages of using water are eliminated.
Eliminates high cost of water including expense of treating water.
Thermal or chemical pollution of water resources is avoided.
Installation is simplified due to elimination of coolant water piping.
Location of the air cooled heat exchangers is independent of water supply location.
Maintenance may be reduced due to elimination of water fouling characteristics which could require frequent cleaning of water cooled heat exchangers
Air cooled heat exchangers will continue to operate (but at reduced capacity) due to radiation and natural convection air circulation should a power failure occur.
Temperature control of the process fluid may be accomplished easily through the use of shutters, variable pitch fan blades, variable speed drives, or, in multiple fan installations, by shutting off fans as required.
Disadvantages
Since air has relatively poor thermal transport properties when compared to water, the air cooled heat exchanger could have considerably more heat transfer surface area. A large space requirement may result.
Approach temperature differences between the outlet process fluid temperature and the ambient air temperature are generally in the range of 10 to 15 °K. Normally, water cooled heat exchangers can be designed for closer approaches of 3 to 5 °K. Of course, closer approaches for air cooled heat exchangers can be designed, but generally these are not justified on an economic basis.
Outdoor operation in cold winter environments may require special consideration to prevent freezing of the tube side fluid or formation of ice on the outside surface.
The movement of large volumes of cooling air is accomplished by the rotation of large diameter fan blades rotating at high speeds. As a result, noise due to air turbulence and high fan tip speed is generated.

This bond may be mechanical or metallurgical in nature. Metallurgical bonds are those, in which a solder, braze, or galvanizing alloy coats the fin and bare tube or in which the fin is welded to the tube. Fins, which are extruded or machined from the base tube and are, therefore, integral with the tube, may also be considered as having a metallurgical type bond. Mechanically bonded tubes may be of two types. First, *imbedded* or *grooved* tubes are formed by machining a helical groove along the length of the tube. The fin is located in the groove and wrapped around the tube, after which the tube material is deformed at the base of the fin. This procedure holds the fin in place and in contact with the tube.

Mechanically bonded tubes may be obtained by mechanically stressing the fin material and/or the tube material to hold the two elements in pressure contact with one another. So called tension wound fins are formed by winding the fin material under tension in a helical manner along the length of the tube.

This method stresses the fin material to maintain contact with the tube. The ends of the fins must be held in place to keep the fins from loosening. This may be done by means of stapling, brazing, soldering, welding or any other way to keep the fins from unwrapping.

Individual fins may be preformed and inserted over the tube, after which the mechanical bond may be obtained by either shrink fitting the fins onto the tube or by expanding the tube radially outward to make pressure contact with the fin material. The means to expand the tube may be hydraulic by pressurizing the tube beyond its yield point; or it may be of a mechanical nature, in which an oversized ball or rod is pushed through the length of the tube, forcing the tube material outward against the fin.

Tubes whose fins are integral with the tube may also be classified as a mechanical bond type, if a liner tube is used inside the finned tube. A liner tube of another material may be used for compatibility with the tube side process fluid. The contact between the two materials could be formed by expanding the liner tube or by drawing the outer finned tube down over the liner. The operating temperatures of the exchanger, including upset or transient conditions may affect the bonding method, which can be used for the finned tubes. In order to maintain design thermal performance, the bond between the fin and the tube must not deteriorate due to a loosening of the fin, which could result from unequal thermal expansion of the fin and tube materials. In order to avoid this degradation of tube performance, mechanically bonded tubes of the tension type are normally limited to temperatures of 400 to 600 °K; and mechanically bonded grooved fin types from 600 to 700 °K. Metallurgically bonded tubes are limited to temperatures below the melting point of the bonding alloy or to a temperature, dependent upon the physical properties of the tube and fin materials.

The operating environment may influence the choice of materials used and the shape of the fin. Aluminum is very often satisfactory as a fin material, although

copper, steel and stainless steel fins are also used. The fin shape may be of edge-type, L-foot type or double L-foot design. The edge type is used for the grooved fin tube, and in cases, where the base tube is not subject to corrosion.

The L-foot fin covers the tube more or less completely to protect the base tube against corrosive attack, but still leaves a potential corrosive site at the base of the fin adjacent to the preceding fin. The double L-foot is intended to provide complete coverage of the tube, where corrosion would otherwise be a problem. Where corrosion is troublesome, soldered or galvanized tubes may offer a solution. The dimensions of finned tubes are results of past experience in the design of air cooled heat exchangers. Tube diameters range from about 1.905 cm (0.75 in.) to 5.08 cm (2.0 in.).

Helically wrapped fins are fabricated such that the fin height can be between about 3/8 to 3/4 of the tube diameter, but limited because of fabrication requirements to a maximum of about 2.54 cm (1.0 in.) in height. Fin spacings vary between about 275 and 450 fins per meter of tube length, while fin thicknesses range from 0.025 to 0.075 cm. For particular cases these parameters may be varied further.

Tube Bundle

A typical tube bundle arrangement is illustrated in Figure 2. The finned tubes are assembled into the tube bundle. Tube lengths range from about 1.83 m long to as much as 12.2 m long. The number of tube rows deep in the bundle is a function of the performance required and generally ranges between 3 and 30. The ends of the tubes are not finned. This permits the tubes ends to be inserted into tubesheets, located at each end of the bundle. The tubesheets separate the cooling air on the fin side from the process fluid on the tube side. Generally, the tube ends are roller expanded into the tube holes in the tubesheet to form the joint, although for higher pressure applications these may be welded joints.

The tubesheets are attached to tube side headers, which contain the tube side fluid and distribute it to the tubes. The headers may be designed to permit any number of tube side passes for the process fluid. For multipass tube bundles, the headers contain partition plates, which divide the bundle into separate passes. However, these may be limited by the operating temperature conditions. If there is a large temperature difference per pass, then the hotter tubes may expand lengthwise to a much greater extent than the tubes in succeeding passes. This could result in high stresses on the tube joint, resulting in leakage at the joint. If differential expansion between passes is excessive, split headers may be necessary. The tube bundle is normally permitted to float independently of the supporting structure due to overall bundle expansion.
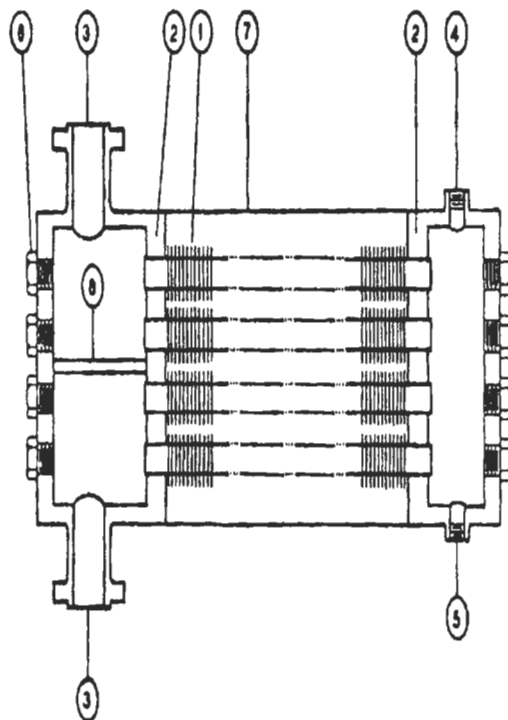
Figure 2. *Typical tube bundle (two pass) using box headers with tube plugs opposite each tube end. Key: (1) Tube; (2) Tube Sheet; (3) Inlet/Outlet Nozzles; (4) Vent; (5) Drain; (6) Tube Plugs; (7) Side Frame; (8) Pass Rib* .

End plates on the tube side headers frequently include removable plugs. These can be pipe tap plugs or straight threads with gasket seals. The plug are located opposite each tube end to permit access for each tube for re-rolling of the tube to tubesheet joint, should leaks, occur and for cleaning the tubes if this should be necessary. If the tubes are welded into the tubesheets and the process fluid conditions are non-fouling, these plugs are not necessary.

An alternate method of providing access to all tubes for repair and cleaning is to use removable bonnet headers. These designs require gaskets to keep the process fluid from leaking to the atmosphere, but may be advantageous for high tube side fouling conditions. Special header designs may be provided for high tube side pressure conditions. These may be circular headers with individual tubes welded in place or billet type headers with flow passages machined into thick steel sections.

The tube bundle is fabricated as a rigid structure to be handled as an individual assembly. Structural steel side members and tube supports are used for this purpose. Such supports are used beneath the bottom of the tubes to prevent the

bundle from sagging; between tube rows to maintain tube spacing and prevent meshing or deformation of the fins; and across the top row of tubes to keep the tubes in proper position. The supports are spaced evenly along the bundle length at intervals, not exceeding about 1.5 meters.

Fan and Drive Assemblies

Fans are used, which correspond to the dimensions of the tube bundle and the performance requirements for the heat exchanger. Normally, the fan diameter is approximately equal to the bundle width, although smaller diameters may be used. For square, or nearly square bundles, one fan is used. For long rectangular bundles, a number of fans operating in parallel may be used. Fans are of axial flow design, which move relatively large volumes of air at low pressure. In order to minimize air recirculation and improve fan efficiency, fan blades are set within orifice rings which provide close radial clearance between the ring and the blade tips. The ring often has a contoured shape to provide a smooth entrance condition for the air. This minimizes air turbulence at this point, which also helps to reduce noise, generated by the fan.

Rotating at high speeds, the fan blades must be balanced to insure that centrifugal forces are not transmitted through the fan shaft to the drive or to the supporting structure. An unbalanced blade could result in severe vibration conditions. Blades are frequently made of aluminum, but other metals and plastics have also been used. Consideration of maximum operating temperature must be given when using the plastic blades. Where corrosion is possible, blades can be coated with epoxies or other suitable protective material. Smaller diameter fans, up to about 1.5 or 2 meters in diameter, can be driven with electric motors. Larger diameter fans are usually indirectly driven by electric motors or steam turbines, using V-belts or gears. V-belt drives are often limited to fan diameters of about 3 meters and less and motors not exceeding 30 hp.

For larger motors and larger diameter fans, right angle gear drives are used. Indirectly driven fans can offer the advantage of speed variation, such that, as the air cooler heat toad varies, the volume of cooling air can also be varied. The fan laws, which relate speed to fan performance show, that reducing speed can also reduce power consumption. The fan may be designed for either forced air flow or induced air flow. In forced-flow installations, the fan blows ambient air across the tube bundle. Induced-draft fans draw the air across the bundle. Therefore, the fan blades are in contact with the heated air, coming off the heat exchanger. This situation gives a power advantage for the forced draft design.

The total pressure of the fan is the sum of the static pressure loss of the air flowing across the tube bundle, plus the velocity pressure of the air, moving

through the fan. Static pressure losses are of the order of 0.5 cm to 3 cm water gauge, while fans are usually designed for velocity pressure of about 0.25 cm water gauge. The actual volumetric flowrate of air, for a given mass flowrate, is directly proportional to the absolute temperature of the air.

Fan efficiencies are typically about 65 % while drive efficiencies are 95 % or better. This power advantage for forced-draft designs generally proves to result in a more economical heat exchanger. Since the fan is close to the ground, structural costs may be less with the drive assembly, located at ground level. However, induced-draft air cooled heat exchangers offer the advantage of better air distribution across the bundle, due to relatively low air velocities approaching the tubes. Furthermore, the air exit velocities of induced-draft heat exchangers are much higher than a forced-draft design. Thus, the possibility of recirculating hot discharge air is less for the induced-draft. When cooling the process fluid to a temperature close to the inlet ambient air temperature, this may be of particular importance.

In a typical air cooled application, impeller air flow is used to cool media, flowing through the banks of heat exchangers. As in many cases, there is only a single air source, and, hence, the design of a heat exchanger effects the other in the heat exchanger bank. A typical example is a radiator/cooler oil package. As the air flow has to take away heat from the radiator and the oil cooler, both must be designed optimally to make the most efficient package. Any over-designing on any of the units, radiator or oil cooler, will adversely effect the performance of the other.
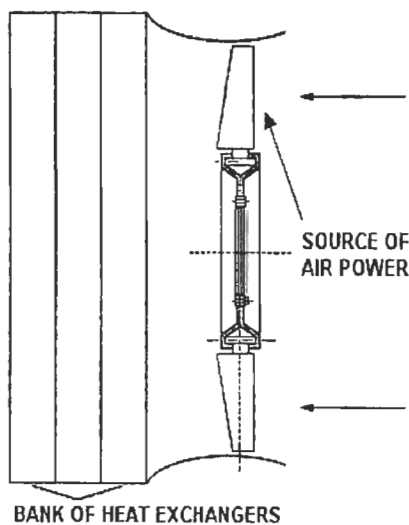
Figure 3. *High-efficiency aerofoil axial impeller.*

As noted earlier, the impeller is a central part of any air cooled heat exchanger. To ensure that the best performance is achieved and power consumption and noise levels are as low as possible, it is important that the proper impeller is selected. Figure 3 shows a multi-wing high-efficiency aerofoil axial impeller. Multi-wing axial impellers can be used in almost any application.

Air Plenum Chamber

The velocity of the air, flowing through the fan, can be as much as 3 to 4 times the velocity across the face of the tube bundle. Also, the air, coming from the circular shape of the fan, must be distributed across the square or rectangular shape of the bundle. The air plenum chamber is intended to make this velocity and shape transition, such that the distribution of air is uniform across the bundle. Common practice is to install the fan in a chamber, such that the distance from the first row of the tube bundle to the fan is about one-half the fan diameter

The plenum chamber design may be a simple box shape, formed by flat sides and bottom, or curved transition sections may be used to obtain a tapered smooth transition from the rectangular bundle to the circular fan. Either design may be used for forced-draft or induced-draft air cooled heat exchangers.

Structural Assembly

The structural assembly of the air cooled heat exchanger is strongly dependent upon the particular plant site requirements of the user. Taken into account should be mechanical loads upon the heat exchanger structure, due to its own weight, of course, but other loadings, such as wind loads, impact loads, nozzles loading and seismic forces must be considered. The presence of equipment beneath the air cooled heat exchangers may require particular designs. Safety considerations may call for fencing or fan guards. Environmental factors could indicate the need for louvers, hail screens, or other protective devices. In addition, the physical location of the heat exchangers may require ladders, platforms, railings, safety cages and other miscellaneous items, which the user will require.

General Exchanger Configurations and Applications

As described above, in the air-cooled exchanger a motor and fan assembly forces ambient air over a series of tubes to cool or condense the process fluids carried within. The tubes are typically assembled in a coiled configuration. Air is

inexpensive and abundant, but it is a relatively poor heat transfer medium. To increase the heat transfer rates of the system, the tubes in air-cooled exchangers are typically given fins, which extend the surface area, increase heat transfer, and give such systems the nickname *fin-tube coils*. Air-cooled exchangers are typically found in such applications, as heating and air conditioning, process heating and cooling, air-cooled process equipment, energy and solvent recovery, combustion air preheating, and fluegas reheating.

The diameter and materials, specified for the tubes and fins, depend on system requirements. The fins are commonly made from aluminum or copper, but may be fabricated of stainless or carbon steel. Tubes are generally copper, but can be made from almost any material, and they range in size from 5/8- to 1-in. outer diameter. The design of the air-cooled exchanger is such, that individual coils can be removed independently for easy cleaning and maintenance. There are several common design configurations that are commercially available. Each is briefly described below.

Aluminum brazed-fin exchangers consist of corrugated plates and fins, which are added to a brazed-composite core to create alternating air and fluid passages. This compact, lightweight design is considered the most cost-effective air cooled unit available. Turbulence created in the fluid channels boosts efficiency. Typical applications include cooling lube oil for power equipment, cooling fluids for hydraulic equipment, and cooling gear box fluids.

Aluminum plate-fin exchangers are constructed with traditional heat exchanger tubing. Stacked, die-formed aluminum plates extend the surface to maximize air-side heat transfer. Like the brazed-fin exchanger, this unit is also used for oil and glycol cooling, but its higher flowrate expands its capabilities. Constructed from standard components, aluminum-fin exchangers are designed with a more solid construction than their brazed-fin counterparts. Typical applications include oil cooling, compressed-air cooling, water cooling with air.

Fin-tube exchangers consist of one continuous fin wrapped spirally around a series of individual tubes. Often referred to as a "heavy duty coil", this air exchanger has fin-tube attachments that can be built either to ASME and API standards, or to customer specifications. Often used in air-heating applications, the heavy-duty coil is available with several different fin variations, including the tapered fin, footed 'V' fin, overlapped-footed fin and the embedded fin, which describe the geometries at the fin-tube interface. The method of attaching the fin to the tube is critical, since the loosening of this bond may hinder heat exchange. Typical applications include those that heat air with high-pressure or high-temperature steam, heating or cooling applications with high liquid flows, cannot tolerate condensate freezing - such as steam applications, and heating air with hot water.

The Fine Wire Heat Exchanger

These types of heat exchanger systems are typically used for indoor climate control. On a flat surface, one has a heat transfer coefficient to air of about 20 W/m^2K. On a fine wire, say a wire of 0.1 mm, one can reach 300 W/m^2K. Fine wires have the same cost per square meter as flat surfaces. This type of air cooled heat exchanger generally uses a ceiling fan that can heat or cool air with only a few degrees °C. The fine wire heat exchanger consists of a woven cloth stacked in parallel strips around a slow turning Sirocco-type fan. This type of fan has applications for space cooling and heating, improves the COP (Coefficient of Performance) of heat pumps and makes small-scale seasonal storage possible.

Let's consider the physics of heat transfer to a fine wire. In doing so, a practical set of issues is to determine the optimal wire diameter (100 μ) at which wire cost/performance is minimal, and come at a heat transfer from air to wire cloth as a function of air velocity. A next step would be to treat the pressure drop through wire cloth, and find the optimal air speed (0.4 m/s), at which the sum of the energy loss of pumping and heat transfer is minimal. With these values we find the optimal temperature drop (2.4°C) over the heat exchanger by minimizing the yearly cost of the exchanger due to investment, and the cost of energy loss due to heat transfer.

A widely used correlation for heat transfer from a cylinder in a perpendicular flow is:

$$Nu = 0.57 \times Re^{0.5} \times Pr^{0.33}$$

$$and \ a = Nu \times l/d$$

When we fill in h=1.85e-5 Pa×s; ρ=1.3 kg/m^3, C_p=1010 J/kg-°K and α=0.025 W/m-°K for the material constants of air at room temperature, this correlation becomes a=3.2×(v/d)$^{0.5}$.

We see that the heat transfer coefficient is inversely proportional to the square root of the wire diameter, which is the reason for the development of fine wire heat exchangers after all. With an air velocity v of 0.5 m/s and a wire of 100 m, we have a=226 W/m^2K, which is around ten times the typical value of flat plate heat exchangers to air.

Fine wires can only be efficiently incorporated into a device using textile technology, such as weaving, and in the case of heat transfer from water to air, one has to weave copper capillaries into copper fine wires. This leads naturally to a cloth where at the capillaries, the wires have a spacing equal to their diameter, and at the wire crossing in the mid point between the capillaries, a zero spacing between the capillaries. This cloth, transferring heat to the air streaming through

it, can be represented as two rows of cylinders, spaced $2 \times d$, in series, perpendicular to the flow. Per square meter there are then l/d wires, each with surface $p \times d$ m^2, so that the heat transfer coefficient related to the cloth surface is $a = p \times 3.2 \times (v/d)0.5 = 10 \times (v/d)^{0.5}$ W/m^2-°K.

The kilogram price of copper fine wire increases with decreasing wire diameter, because of the wire drawing cost. If P_c is the copper wire price per kg, and the density of copper is 8900 kg/m^3, then per square meter cloth there is $l/d \times p/4 \times d^2 \times 8900 \times P_c$ or $7e3 \times d \times P_c$ nlg copper. Per unit of heat transfer coefficient, $10 \times (v/d)^{0.5}$ W/m^2-°K, there is $700 \times d^{1.5} \times v^{-0.5} \times P_c$ nlg copper. So we have the economically optimal wire diameter, when the product $Pe \times d^{1.5}$ is minimal. This is the case with a wire diameter of 100 mm. With this optimal wire diameter, the calculated heat transfer coefficient, related to the cloth, surface is $1000 \times v^{0.5}$ W/m^2-°K.

The following correlation for pressure drop describes flow perpendicular to pipe bundles for Re $< 25 \times x/(x-1)$, where x is the ratio of pipe spacing to pipe diameter, v means the mean fluid velocity over the bundle front surface, l - the pipe bundle length in the flow direction, and d - the pipe diameter. In our case, we have two bundles in series with $x = 2$ and with $l/d = 1$ at a distance of more than $2 \times d$, and $d = d = 1e-4$ m, so we can write here for the pressure drop:

$$\Delta P = 2 \times 28.4/p \times h/r/v/d \times 1 \times 0.5 \times r \times v^2 ;$$

$$\text{or } \Delta P = 6.6 \times v, \text{ for } Re < 25 \text{ or } v < 3.8 \text{ m/s}$$

By blowing air through the cloth we have to use energy, or electric energy. When the ceiling ventilator motor has a constant efficiency of $6W_{mech} / 45 W_{el}$, and the total pressure drop, inclusive the acceleration term, through the fan $\Delta P = 6.6 \times v + 0.5 \times v^2$, the surface of the heat exchanger is A, then we need $45/6 \times \Delta P \times A$ electric power to move the air.

By moving the air faster, we increase the heat transfer, and so save energy by lowering the temperature drop through which the heat flows. We can express this energy by estimating the electric energy, we need to pump heat from outside air at a mean temperature over an arbitrary heating season of 4.8 °C to heat inside air at a mean temperature of 17.3 °C.

For each increase of the delta temperature of one of the heat exchangers by 1 °C, we need an extra $F_w /(17.3-4.8)/ 8$ amount of electric power when the COP of a heat pump is 8. When we use the heat transfer and pressure drop equations in these functions for a heat exchanger of $F_w = 1$kW, the sum of the energy losses is:

$$45/6 \times A \times (0.5 \times v^2 + 6.6 \times v) + (1kW)^2 /8/12.5/ 1000 \times v^{0.5} \times A, \text{ or}$$

$$A \times (3.75 \times v^2 + 49.5 \times v + 10 \times v^{-0.5})$$

The value of v, at which this function has its minimum value, is 0.21 m/s. In practice, the efficiency of a standard one-phase fan motor increases with its output, and this increases the optimum air velocity to about v = 0.4 m/s in a typical case. This low optimal air speed is the reason that the heat exchanger surface should be plied in a zig-zag fashion to increase the frontal air speed to a more acceptable value of about 2 m/s, in order to keep the apparatus in a compact form.

When we take v to be 0.4 m/s, our cloth surface at 1 kW becomes

$$A = 1e^3 / 1000.0.4^{0.5}/\Delta T = 1.6 / \Delta T \text{ m}^2(\text{nlg}),$$

or when we write the cloth cost of Pa off in 7 years, $1.6/7 \times Pa/\Delta T$ nlg/y.

The energy loss is $1kW \times \Delta T/12.5/COP = 10 \times \Delta T$ (W), this power during the heating season of 5080 hours, $10 \times \Delta T \times 5080 = \Delta T \times 50.8$ kWh/y, or with a kWh price at 0.2 nlg, $10.2 \times \Delta T$ nlg/y. The sum of capital cost and energy loss cost is $0.23 \times Pa/DT + 10.2 \times \Delta T$. With Pa = 300 nlg/m^2 cloth, mounted in a heat exchanger, this function is minimal with Pa = 300 nlg/m^2, at T=2.6 °C, and with Pa = 200 nlg/m^2, at ΔT= 2.2 °C. The cost price is one of the last parameters that becomes known, but a value for the optimal ΔT = 2.5 °C can be taken. This in sharp contrast with usual tube-plate-fin heat exchangers, that have optimal temperature drops of 15 to 20 °C.

In home or building heating applications, a slowly turning fan drives air (typically 500 liters per second) through a maze of copper fine wire gauze, woven around water carrying copper capillaries: a fine wire water-to-air heat exchanger. The result is one million 100μ, 0.6 mm long copper fins that transfer heat ten times better than a typical central heating radiator. The performance can be described with the following formulae in Table 2, where D = diameter fan in m; n = speed of fan in RPM.

Table 2. *Formulae for Fine Wire Heat Exchanger*

Parameter	Formula	Units
Heat transfer	$8 \times D^2 \times n^{0.8}$	W/°C
Fan power (Pm)	$3 \times 10^{-5} \times D^4 \times n^3$	W
Noise level	$20 \times \log Pm + 28$	dB(A)
Cost in series of 1000	$280 \times D$	U.S. \$

A fan with a diameter of 60 cm and a speed of 100 RPM will have a heat transfer capacity of 100W/°C, needs a mechanical power of 4W, has a noise level of 40 dB(A) and costs 180 Euro.

Using this type of heat exchanger fan, rooms and working spaces can be cooled with ground water, that is only about 3 to 5 degrees cooler, than the desired room temperature. A leading European manufacturer of these types of heat exchanger systems is Fiwihex in The Netherlands, where ground water is normally about 12 °C. Hence, one 100 W/°C ceiling fan can cool a room to 20 °C with a power of $(23-12) \times 100 = 1100$ Watt. This is about the power (3750 BTU/hr) that a portable air conditioner can deliver. As an example, when we need this 100 W/°C $= 8 \times D^2 \times n^{0.8}$, select a fan with a D of 60 cm. The speed n becomes then $(2000/(20-12)/8/0.6^2)^{(1/0.8)} = 84$ rpm, the mechanical power becomes $3 \times 10^{-5} \times 0.6^4 \times 84^3 = 2.3$ Watt, and the noise level becomes 20 log 2.3 + 28 = 35 dBA. This is a level of noise that is so low that it is never reached inside a city environment. The fan and water circulation pump will use about 60W of electricity, this has to be compared with 400W for a typical air conditioner.

The heat exchangers described are so powerful that heat extraction from the outside air is now an economic possibility, when no ground or surface water is available. A Coefficient Of Performance of 6 is attainable as a mean over the Dutch heating season (inside temperature 20 °C, outside 4.8 °C) according to Fiwihex, with the following configuration: 2×1000 W/°C propane-to water heat exchangers; 2×500 W/°C fans and a standard shop display refrigeration compressor. The heat pump stops during electricity peak hours when the heat storage system has been installed. When (ground)water as a heat source is available, the COP rises to 8.

SHELL AND TUBE TYPE HEAT EXCHANGERS

Tubular Exchanger Manufacturers Association (TEMA)

The Tubular Exchanger Manufacturers Association, or TEMA, is a group of leading manufacturers, who have pioneered the research and development of heat exchangers for over fifty-five years. Founded in 1939, TEMA has grown to include a select group of member companies. Although it may be easy to choose a TEMA member as a supplier, it is not easy for manufacturers to become a TEMA member. Member companies must meet stringent criteria to even qualify for TEMA membership, and are periodically examined by TEMA to ensure that the manufacturer meets membership criteria, and designs and manufacturers according to TEMA standards. Members adhere to strict specifications. TEMA Standards and Software have achieved worldwide acceptance as the authority on shell and tube heat exchanger mechanical design. These tools give engineers a

valuable edge when designing and manufacturing all types of heat exchangers. Seven editions of TEMA Standards have been published, each on updating the industry on the latest developments in technology. TEMA has also developed engineering software that complements the TEMA Standards in the areas of flexible shell elements (expansion joints) analysis, flow induced vibration analysis and fixed tubesheet design and analysis. This state-of-the-art software works on an IBM PC or compatible, and features a materials data-bank of 38 materials, as well as user-friendly, interactive input and output screens. The programs handle many complex calculations, so users can focus on the final results. Many companies can manufacture heat transfer equipment, but not all can assure its safe, effective design and quality construction. That's why the TEMA Heat Exchanger Registration System was instituted in 1994. For quality assurance, one need only look for the TEMA Registration Plate attached to the heat exchanger. Each plate includes a unique TEMA registration number. Before a company can even become a member of TEMA and participate in the registration system, it must have a minimum of 5 years of continuous service in the manufacture, design and marketing of shell and tube heat exchangers.

All TEMA companies must have in-house thermal and mechanical design capabilities, and thoroughly understand current code requirements and initiate strict quality control procedures. Additionally, all welding must be done by the company's own personnel, and the company must have its own quality control inspectors. These criteria ensure the highest level of technical expertise, which gives TEMA members a meaningful advantage when designing or fabricating heat exchangers. The following is a list of TEMA manufacturers.

Alco Products - 8505 Jacksboro Highway, Wichita Falls, TX 76302-9703 (Phone: 940-723-6366 Fax: 940-723-1360)

API Heat Transfer Inc. - 2777 Walden Ave, Buffalo, NY 14225 (Phone: 716-684-6700 Fax: 716-684-2129)

Cust-O-Fab, Inc. - 8888 W 21st St, Sand Springs, OK 74063 (Phone: 918-245-6685 Fax: 918-241-1434)

Energy Exchanger Co. - 1844 North Garnett Road, Tulsa., OK 74116 (Phone: 918-437-3000 Fax: 918-437-7144)

Engineers & Fabricators Co. - 3501 West 11th Street, Houston, TX 77008-6001 (Phone: 713-803-4700 Fax: 713-869-8088)

Fabsco Shell & Tube, LLC - P.O. Box 988, Sapulpa, OK 74066 (Phone: 918-224-7550 Fax: 918-224-3564)

Graham Corporation - 20 Florence Avenue, Batavia, NY 14020 (Phone: 716-343-2216 Fax: 716-343-1097)

Heat Transfer Equipment Co. - P.O. Box 580638, Tulsa, OK 74158 (Phone: 918-836-8721 Fax: 918-838-3570)

Hughes-Anderson Heat Exchangers, Inc. - 1001 N. Fulton Ave, Tulsa, OK 74115 (Phone: 918-836-1681 Fax: 918-836-5967)

ITT Standard - 175 Standard Parkway, Buffalo, NY 14240 (Phone: 716-897-2800 Fax: 716-897-1777)

Joseph Oat Corporation - 2500 Broadway, Camden, NJ 08104 (Phone: 856-541-2900 Fax: 856-541-0864)

Manning & Lewis Eng. Co. - 675 Rahway Ave, Union, NJ 07083 (Phone: 908-687-2400 Fax: 908-687-2404)

Nooter Corporation - P.O. Box 451, St, Louis, MO 63166 (Phone: 314-421-7561 Fax: 314-421-7580)

Ohmstede, Inc. - St. Gabriel, Louisiana 70776 (Phone: 409-833-6375 Fax: 409-833-6735)

R.A.S. Process Equipment, Inc. - 324 Meadowbrook Road, Robbinsville, NJ 08691 (Phone: 609-371-1000 Fax: 609-371-1221)

Southern Heat Exchanger, Corp. - P.O. Box 1850, Tuscaloosa., AL 35403 (Phone: 205-345-5335 Fax 205-345-0009)

Steeltek, Inc. - 4141 South Jackson, Tulsa, OK 74107 (Phone: 918-446-4001 Fax: 918-446-8317)

Struthers Industries,Inc. - 1500 34th Street, Gulfport, MS 39502 (Phone: 228-864-5410 Fax: 228-864-5555)

Wiegmann & Rose - (Xchanger Manufacturing Corp.) P.O. Box 4187, Oakland, CA 94614 (Phone: 510-632-8828 Fax: 510-632-8920)

Yuba Heat Transfer - (div. of Connell Ltd, Partnership) P.O. Box 3158, Tulsa, OK 74101 (Phone: 918-234-6000 Fax: 918-234-3345)

The greatly expanded 8th Edition of the Standards of the Tubular Exchanger Manufacturers Association retains the useful data and features, found in the Seventh Edition, plus many clarifications and innovations. All sections have been reviewed to incorporate new data, which were not available at the time of the 1988 printing, including suggestions, which resulted from the extensive use of the Standards by both manufacturers and users of shell and tube heat exchangers. Many helpful recommendations were also received through the cooperation of the American Petroleum Institute (API) and the American Society of Mechanical Engineers (ASME). Some noteworthy features of the Eighth Edition include: **(a)** Metrification has been included where feasible and appropriate; **(b)** Methods for calculating several types of floating head backing rings have been added; **(c)** A method for incorporating pass partition rib area into flange design has been incorporated; **(d)** The vibration section has been expanded and vibration amplitude for vortex shedding and acoustic resonance have been added; **(e)** Nozzle flange pressure/temperature rating tables from ASME Standard B16.5-1996 w/ 1998 addenda are included; **(f)** New materials have been included in coefficient of thermal expansion, modulus of elasticity, and thermal conductivity tables; **(g)** Design equations for double tubesheets have been added; **(h)** A method for calculating the mean metal temperature for tubesheets has been added; **(i)** Stress multipliers have been added to account for the stiffness of

knuckles on flanged and flued expansion joints; **(j)** Suggested calculation methods have been incorporated for both vertical and horizontal supports; **(k)** Design methods have been added for lifting lugs; **(l)** A demonstration copy of the available software is included with the purchase of each Standard.

The Tubular Exchanger Manufacturers Assn. has established heat exchanger standards and nomenclature. Every shell-and-tube device has a three-letter designation; the letters refer to the specific type of stationary head at the front end, the shell type, and the rear-end head type, respectively (a fully illustrated description can be found in the TEMA standards). Common TEMA designations are listed with specific configurations described below.

Shell and Tube Configurations

The shell and tube heat exchanger consists of a shell, usually a circular cylinder, with a large number of tubes, attached to an end plate and arranged in a fashion where two fluids can exchange heat without the fluids, coming in contact with one another. The most common types of heat exchangers configurations are illustrated in Figure 4.
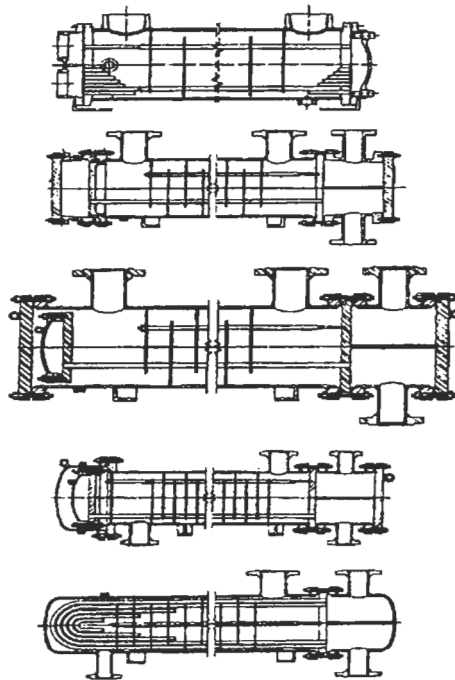
Figure 4. *Common shell-and-tube exchanger configurations.*

There are many text books that describe the fundamental heat transfer relationships, but few discuss the complicated shell side characteristics. On the shell side of a shell and tube heat exchanger, the fluid flows across the outside of the tubes in complex patterns. Baffles are utilized to direct the fluid through the tube bundle and are designed and strategically placed to optimize heat transfer and minimize pressure drop.

A measure of the complexity of predicting shell side heat transfer can be obtained by considering the path of shell side fluid flow. The flow is partially perpendicular and partially paralleled to the tubes. It reverses direction as it travels around the baffle tips and the flow regime is governed by tube spacing, baffle spacing and leakage flow paths. Throughout the fluid path, there are a number of obstacles and configurations, which cause high localized velocities.

These high velocities occur at the bundle entrance and exit areas, in the baffle windows, through pass lanes and in the vicinity of tie rods, which secure the baffles in their proper position. In conjunction with this, the shell side fluid generally will take the path of least resistance and will travel at a greater velocity in the free areas or by-pass lanes, than it will through the bundle proper, where the tubes are on a closely spaced pitch. All factors considered, it appears a formidable task to accurately predict heat transfer characteristics of a shell and tube exchanger.

The problem is further complicated by the manufacturing tolerances or clearances that are specified to allow assembly and disassembly of the heat exchanger. It is improbable that these clearances will all accumulate to either the positive or negative side, so it is customary to compute heat transfer relationships on the basis of average clearances.

The various paths of fluid flow through the shell side of a segmental baffle heat exchanger is illustrated in Figure 5, whereby the letter designations in the figure are: (A) leakage stream through the annular spaces between tubes and baffle holes of one baffle; (B) cross flow stream through the heat transfer surface between successive baffle windows. It will be noted that this stream is made of B_1 (a portion of fluid passing through baffle windows) plus portions of the A stream; (C) by-pass stream on one side of tube nest flowing between successive baffle windows; (D) leakage stream between shell and edge of one baffle. The by-pass area C between the bundle and shell can be reduced by using dummy tubes, seating strips, or tie rods with seal strip baffles.

The dummy tubes do not pass through the tubesheets, and can be located close to the inside of the shell. The seating strips extend from baffle to baffle in a longitudinal direction and effectively channel the fluid across the tubes to minimize turbulence and heat transfer. On some fixed tubesheet designs, the outer tubes are in close proximity to the inside of the shell so that by-pass is minimal and no by-pass elimination is necessary. There are a number of

techniques that can be employed to reduce the flow in areas A and E. Tight tolerances are often employed and some manufacturers use a punched collar baffle where the tube holes in the baffle have a small precision collar which minimizes clearances between tube and tubehole with the added benefit of good tube support. The baffles are sometimes welded at the shell's periphery to completely eliminate by-pass. Each of these techniques is effective, but are governed by the trade off of increased efficiency versus added cost.

Shell and tube heat exchangers are generally designed with a certain degree of conservatism from both the thermal and mechanical design aspects. From a thermal design viewpoint, the conservatism arises from excessive surface to accommodate fouling in service. From a mechanical design viewpoint, design procedures generally employ allowable stresses, which are based on a factor of safety. But, even so, shell and tube heat exchangers experience problems in service. One of these problems concerns fouling of either the tube side or shell side of the heat exchanger.

Fouling is an accumulation of scale or dirt on the tube surface, thereby adding a resistance to heat transfer. It is very difficult to accurately predict the degree of fouling for a specified service period. There are minimal documented test results on this subject and the results are seldom applicable because of the number of variables in a fouling study. It is, indeed, a fortunate user, who can rely on past performance of the same or similar equipment and specify the proper amount of excess surface required to offset the amount of fouling. For most applications, the degree of fouling is strictly an estimate and the probability is that the heat exchanger is either inadequate or over surfaced. Once the tubes are fouled, they can be either mechanically or chemically cleaned.
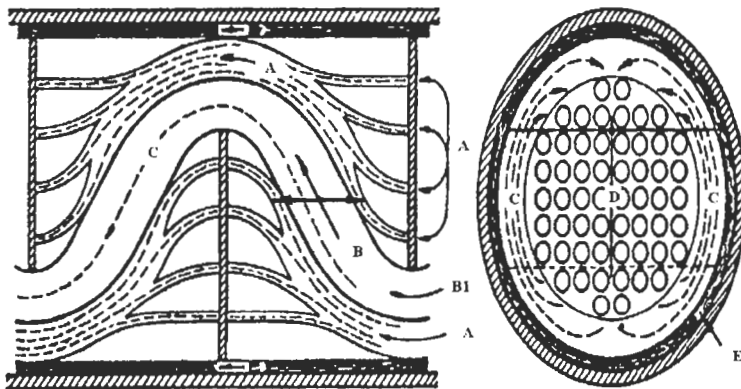

Figure 5. *Illustrates leakage path streams.*

Generally, the tube side presents no particular problem and straight tubes can be easily wire brushed. U-tubes are difficult to clean mechanically and are generally used, where fouling is expected to be minimal. The shell side of the heat exchanger is more difficult to clean, particularly for closely spaced staggered types of tube bundles. Many users specify square or rectangular pitch tube arrangement and removable bundle construction where excessive shell side fouling is expected.

Another serious problem in heat exchangers is corrosion. Severe corrosion can and does occur in tubing and very often with common fluids such as water. Proper material selection based on a full analysis of the operating fluids, velocities and temperatures is mandatory. Very often, heavier gauge tubing is specified to offset the effects of corrosion, but this is only a partial solution. This should be followed by proper start-up, operating and shut-down procedures.

Many heat exchangers use water on the tube side as the cooling medium and compatible copper alloy tubing and still experience corrosion problems. Invariably, this can be traced to some part of the cycle, where the water was stagnant or circulated at extremely low velocity. Most problems with heat exchangers occur during initial installation or shortly thereafter. Improper installation or misalignment can create excessive stresses in supports or nozzles or cause damage to expansion joints or packed joints.

On initial start-up and shut-down the heat exchanger can be subjected to damaging thermal shock, overpressure or hydraulic hammer. This can lead to leaky tube-to-tubesheet joints, damaged expansion joints or packing glands because of excessive axial thermal, expansion of the tubes or shell. Excessive shell side flowrates during the "shake down" can cause tube vibrations and catastrophic failure.

Table 3 provides recommended start-up and shut-down procedures. Effort should be made to avoid subjecting units to thermal shock, overpressure, and/or hydraulic hammer, since these conditions may impose stresses, that exceed the mechanical strength of the unit or the system in which it is installed, which may result in leaks and/or other damage to the unit or entire system.

Some general considerations to bear in mind are: (1) In all start-up and shut-down operations, fluid flows should be regulated so as to avoid thermal shocking the unit, regardless of whether the unit is of either a removable or non-removable type of construction; (2) For fixed tubesheet (i.e., non-removable bundle) type units, where the tube side fluid cannot be shut down, it is recommended that both a bypass arrangement be incorporated in the system, and the tube side fluid be bypassed before the shell side fluid is shut down; (3) Extreme caution should be taken on insulated units where fluid flows are terminated and then restarted. Since the metal parts could remain at high temperatures for extended periods of time, severe thermal shock could occur.

Table 3. *Recommended General Start-Up and Shut-Down Procedures*

Fluid Location and Relative Temp.				Start-Up Procedure	Shut-Down Procedure
Shell Side		Tube Side			
Fluid	Temp.	Fluid	Temp.		
Fixed Tubesheet (Non-Removable Bundle)					
L	H	L	C	Start both fluids gradually at the same time.	Shut down both fluids gradually at same time.
Cond. Gas	H	L or G	C	Start hot fluid first, then slowly start cold fluid. Avoid temperature shock.	Shut down cold fluid first, then hot fluid.
G	H	L	C	Start cold fluid first, then hot fluid.	Shut down cold fluid gradually, then hot fluid.
L	C	L	H	Start both flows gradually at the same time.	Shut down both fluids gradually at same time.
L	C	G	H	Start cold fluid first, then hot fluid.	Shut down hot fluid first, then cold fluid.
U-Tube/ Packed Floating Head/ Packed Floating Tubesheet/ Internal Floating Head					
(All these types have removable bundles)					
L	H	L	C	Start cold fluid first, then gradually start hot fluid.	Shut down hot fluid first, then cold fluid.
Cond. Gas	H	L or G	C	Start cold fluid first, then gradually start hot fluid.	Shut down cold fluid first, then gradually shut down hot fluid.
G	H	L	C	Start cold fluid first, then gradually start hot fluid.	Shut down hot fluid first, then cold fluid.
L	C	L	H	Start cold fluid first, then gradually start hot fluid.	Shut down hot fluid first, then cold fluid.
L	C	G	H	Start cold fluid first, then gradually start hot fluid.	Shut down hot fluid first, the cold fluid.

C - Cold; H - Hot; G - Gas; L- Liquid; Cond. Gas - Condensing Gas (e.g., steam)

The shell-and-tube exchanger's flexible design, high pressure and temperature capabilities, and its ability to handle high levels of particulate material make it the most common heat exchanger used in the CPI. Mechanically simple in design and relatively unchanged for more than 60 years, the shell-and-tube offers a low-cost method of heat exchange for many process operations. The most common shell-and-tube configurations are briefly described.

Straight-Tube, Fixed-Tubesheet Exchangers

The fixed-tubesheet exchanger is the most common, and generally has the lowest capital cost per ft^2 of heat-transfer surface area. Fixed-tubesheet exchangers consist of a series of straight tubes sealed between flat, perforated metal tubesheets.

Because there are neither flanges, nor packed or gasketed joints inside the shell, potential leak points are eliminated, making the design suitable for higher-pressure or potentially lethal/toxic service. However, because the tube bundle cannot be removed, the shellside of the exchanger (outside the tubes) can only be cleaned by chemical means.

The inside surfaces of the individual tubes can be cleaned mechanically, after the channel covers have been removed. The fixed-tubesheet exchanger is limited to applications where the shellside fluid is non-fouling; fouling fluids must be routed through the tubes. Common TEMA designations for the straight-tube, fixed-tubesheet exchangers are BEM, AEM, NEN. Common applications include vapor condensers, liquid-liquid exchangers, reboilers, and gas coolers.

Removable-Bundle, Externally Sealed, Floating-Head Exchanger

Floating-head exchangers are so named because they have one tubesheet that is fixed relative to the shell, and another that is attached to the tubes, but not to the shell, so it is allowed to "float" within the shell. Unlike fixed-tubesheet designs, whose dimensions are fixed at a given dimension relative to the shell wall, floating-head exchangers are able to compensate for differential expansion and contraction between the shell and the tubes. Since the entire tube bundle can be removed, maintenance is easy and relatively inexpensive. The shellside surface can be cleaned by either steam or mechanical means. In addition to accommodating differential expansion between the shell and tubes, the floating tubesheet keeps shellside and tubeside process fluids from intermixing. Although the externally sealed, floating-head design is less costly than the full, internal-

floating-head exchanger, it has some design limitations: both shellside and tubeside fluids must be non-volatile or non-toxic, and the tubeside arrangements are limited to one or two passes. In addition, the packing used in this exchanger limits design pressure and temperature to 300 psig and 300 °F. Common TEMA designations are AEW and BEW. Applications include exchangers handling inter- and after-coolers, oil coolers, and jacket water coolers.

Removable- Bundle, Internal-Clamp-Ring, Floating-Head Exchanger

This design is useful for applications where high-fouling fluids require frequent inspection and cleaning. Because the exchanger allows for differential thermal expansion between the shell and tubes, it readily accommodates large temperature differentials between the shellside and the tubeside fluids. This design has added versatility, however, since multi-pass arrangements are possible. However, since the shell cover, clamp ring, and floating-head cover must be removed before the tube bundle can be removed, service and maintenance costs are higher than in "pull through" designs (discussed below). Common TEMA designations are AES and BES. Typical applications include process-plant condensers; inter- and after-cooler designs, gas coolers and heaters, and general-purpose industrial heat exchangers.

Removable-Bundle, Outside-Packed, Floating-Head Exchanger

This design is well suited for applications where corrosive liquids, gases, or vapors are circulated through the tubes, and for air, gases, or vapors in the shell. Its design also allows for easy inspection, cleaning, and tube replacement, and provides large bundle entrance areas without the need for domes or vapor belts. Only shellside fluids are exposed to packing, allowing high-pressure, volatile, or toxic fluids to be used inside the tubes. The packing in the head does, however, limit design pressure and temperatures. Common TEMA designations are BEP and AEP. Common applications include oxygen coolers, volatile or toxic fluids handling, and gas processing.

Removable-Bundle, Pull-Through, Floating-Head Exchangers

In the pull-through, floating-head design, the floating-head cover is bolted directly to the floating tubesheet. This allows the bundle to be removed from the shell without removing the shell or floating-head covers, which eases inspection

and maintenance. This is ideal for applications that require frequent cleaning. However, it is among the most expensive designs. The pull-through design accommodates a smaller number of tubes in a given shell diameter, so it offers less surface area than other removable-bundle exchangers.

Common TEMA designations are AET and BET, and typical applications include exchangers handling chemical fluids, hydrocarbon fluid condensers, air or gas compressors, and inter-and after-coolers.

Removable-Bundle, U-Tube Exchangers

In the U-tube exchanger, a bundle of nested tubes, each bent in a series of concentrically tighter U-shapes, is attached to a single tubesheet, as illustrated in Figure 7. Each tube is free to move relative to the shell, and relative to one another, so the design is ideal for situations that accommodate large differential temperatures between the shellside and the tubeside fluids during service. Such flexibility makes the U-tube exchanger ideal for applications that are prone to thermal shock or intermittent service.

As with other removable-bundle exchangers, the inside of the shell, and to the outside of the tubes. However, unlike the straight-tube exchanger, whose tube internals can be mechanically cleaned, there is no way to physically access the U-bend region inside each tube, so chemical methods are required for tubeside maintenance.
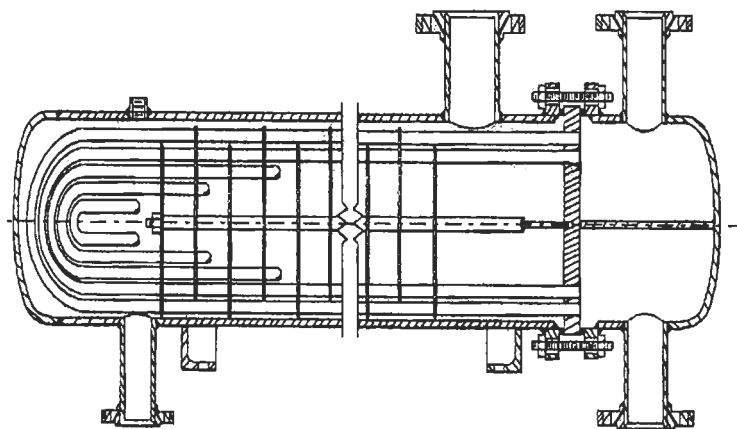
Figure 7. U-tube design.

As a rule of thumb, non- fouling fluids should be routed through the tubes, while fouling fluids should be reserved for shellside duty. This inexpensive exchanger allows for multi-tube pass arrangements. However, because the U-tube cannot be made single pass on the tubeside, true countercurrent flow is not possible. Common TEMA designations are BEU and AEU, and typical applications include oil cooling, chemical condensing, and steam heating applications.

Special Designs

For applications with high vapor-flow and high- pressure conditions, a specially designed shell-and-tube exchanger must often be employed. Special designs may also be called for when applications have close temperature crossings, meaning the outlet temperature of the warmed fluid exceeds that of the cooled fluid.

The following are several examples: TEMA K-type shells, which allow for proper liquid disengagement for reboilers; TEMA J-type shells, which accommodate high vapor flows by allowing for divided flow in the shellside; Two-pass TEMA F-type shells, which can be used for applications when a temperature cross exists (below); TEMA D-type front head designs, which are often the answer for high-pressure tubeside applications.

While these specially designed exchangers may be the solution to a process problem, construction costs tend to be higher than those of "standard" engineered shell-and-tube equipment. Common TEMA designations include BKU, BJM, BFM and DED. Specially designed exchangers are often called for in such applications as reboilers, steam heaters, vapor condensers, and feedwater heaters.

Off-the-Shelf Exchangers

Fixed-tubesheet and U-tube shell-and-tube exchangers are the most common types of off-the-shelf heat exchangers available. Such stock models are typically used as components in vapor condensers, liquid-liquid exchangers, reboilers and gas coolers. Standard fixed-tubesheet units, the most common shell-and-tube heat exchangers, range in size from 2 to 8 in. dia. Materials of construction include brass or copper, carbon steel, and stainless steel. Even though this exchanger is one of the least expensive available, it is still generally constructed to standards specified by the manufacturer. Stock exchangers can be constructed to American Society of Mechanical Engineers (ASME) codes.

U-tube heat exchangers are commonly used in steam heating applications, or heating and cooling applications that handle chemical fluids as opposed to water.

While the U-tube is generally the lowest-priced heat exchanger available, higher service and maintenance costs tend to be higher than other exchangers, since the nested, U-bend design makes individual tube replacement difficult. Custom-designed heat exchangers, though more expensive than their off-the-shelf counterparts, are generally made to higher design standards than stock exchangers.

Many manufacturers follow the TEMA standards for design, fabrication and material selection. TEMA B is the most common TEMA designation, and provides design specifications for exchangers used in chemical process service. TEMA C guidelines provide specifications for units used in commercial and general process applications. TEMA R guidelines provide specifications for exchangers used in petroleum refining and related process operations. Each of these classes are applicable to shell-and-tube heat exchangers with the following limitations: (1) Shell diameter does not exceed 60 in.; (2) Pressure does not exceed 3,000 psi; (3) The product shell diameter (in.) times pressure (psi) does not exceed 60,000. Standards set by the American Petroleum Institute (API; Washington, DC) are also generally accepted throughout the heat exchanger industry. These standards (API 614,660 and 661) specify the mechanical design of the exchanger and list specific materials that can be used in construction of both water-and air-cooled exchangers. While there are significant advantages to purchasing a custom-designed exchanger that meets either TEMA or API manufacturing guidelines, these specifications add to the cost of the exchanger and may slow delivery time.

SPIRAL-PLATE HEAT EXCHANGERS

A spiral-plate exchanger is fabricated from two relatively long strips of plate, which are spaced apart and wound around an open, split center to form a pair of concentric spiral passages. Spacing is maintained uniformly along the length of the spiral by spacer studs welded to the plate.

For most services, both fluid-flow channels are closed by welding alternate channels at both sides of the spiral plate (Figure 8). In some applications, one of the channels is left completely open on both ends and the other closed at both sides of the plate (Figure 9). These two types of construction prevent the fluids from mixing.

Spiral-plate exchangers are fabricated from any material that can be cold worked and welded. Materials commonly used include: carbo steel, stainless steel, nickel and nickel alloys, titanium, Hastelloys, and copper alloys. Baked phenolic-resin coatings are sometimes applied. Electrodes can also be wound into the assembly to anodically protect surfaces against corrosion.

Spiral-plate exchangers are normally designed for the full pressure of each passage. The maximum design pressure is 150 psi because the turns of the spiral are of relatively large diameter, each turn must contain its design pressure, and plate thicknesses are somewhat limited. For smaller diameters, however, the design pressure may sometimes be higher. Limitations of the material of construction govern design temperatures.
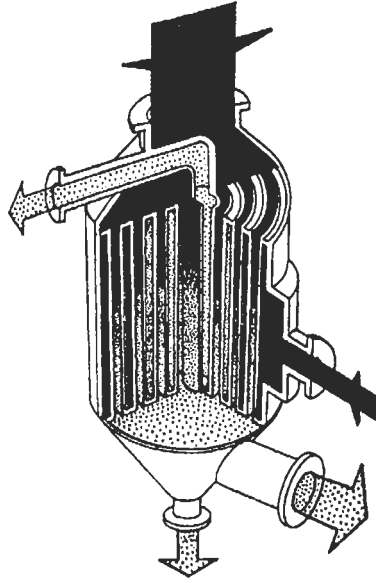

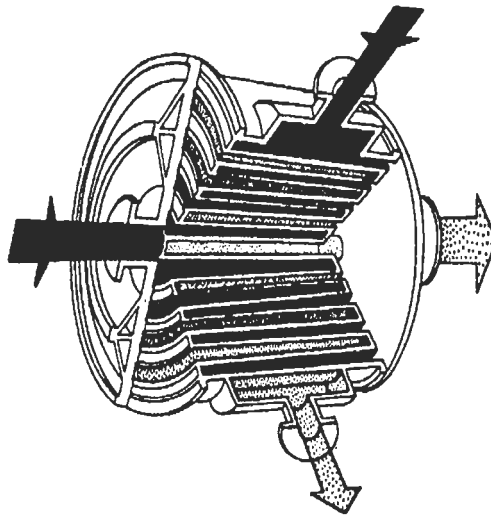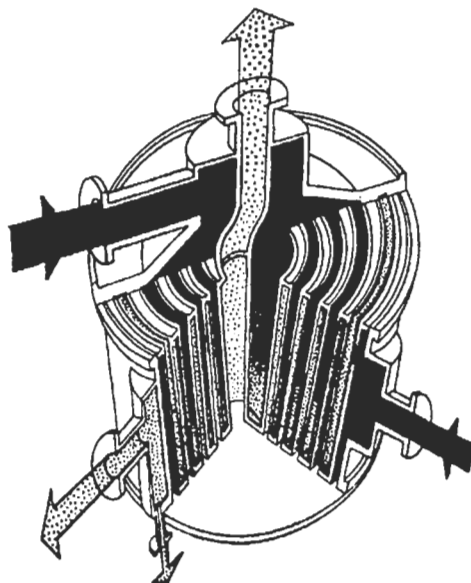
Figure 8. *Flow are both spiral and axial.*

Figure 9. *Spiral flow in both channels.*

**Figure 10.** *Combination flow used to condense vapors.*

The spiral assembly can be fitted with covers to provide three flow patterns: (1) both fluids in spiral flow, (2) one fluid in spiral flow and the other in axial flow across the spiral, and (3) one fluid in spiral flow and the other in a combination of axial and spiral flow.

For spiral flow in both channels, the spiral assembly includes flat covers at both sides (Figure 10). In this arrangement, the fluids usually flow counter-currently, with the cold fluid entering at the periphery and flowing toward the core, and the hot fluid entering at the core and flowing toward the periphery. For this arrangement, the exchanger can be mounted with the axis of the spiral either vertical or horizontal. This arrangement finds wide application in liquid-to-liquid service, and for gases or condensing vapors if the volumes are not too large for the maximum flow area of 72 square inches.

For spiral flow in one channel and axial flow in the other, the spiral assembly includes conical covers, dished heads, or extensions with flat covers. In this arrangement, the passage for axial flow is open on both sides and the spiral flow channel is sealed by welding on both sides of the plate. Exchangers with this arrangement are suitable for services in which there is a large difference in the volumes of the two fluids. This includes liquid-liquid service, heating or cooling gases, condensing vapors, or boiling liquids. Fabrication can provide for single pass or multipass on the axial-flow side. This arrangement can be mounted with the axis of the spiral either vertical or horizontal. It is usually vertical for

condensing or boiling. For combination flow, a conical cover distributes the axial fluid to its passage (Figure 11). Part of the open spiral is closed at the top, and the entering fluid flows only through the center part of the assembly. A flat cover at the bottom forces the fluid to flow spirally before leaving the exchanger. This type is most often used for condensing vapors and is mounted vertically. Vapors flow first axially until the flow volume is reduced sufficiently for final condensing and subcooling in spiral flow.

A modification of combination flow is the column-mounted condenser. A bottom extension is flanged to mate the column flange. Vapor flows upward through a large central tube and then axially across the spiral, where it is condensed. Subcooling may be achieved by falling-film cooling or by controlling a level of condensate in the channel. In the latter case, the vent stream leaves in spiral flow and is further cooled. The column mounted condenser can also be designed for updraft operation and allows condensate to drop into an accumulator with a minimum amount of subcooling.
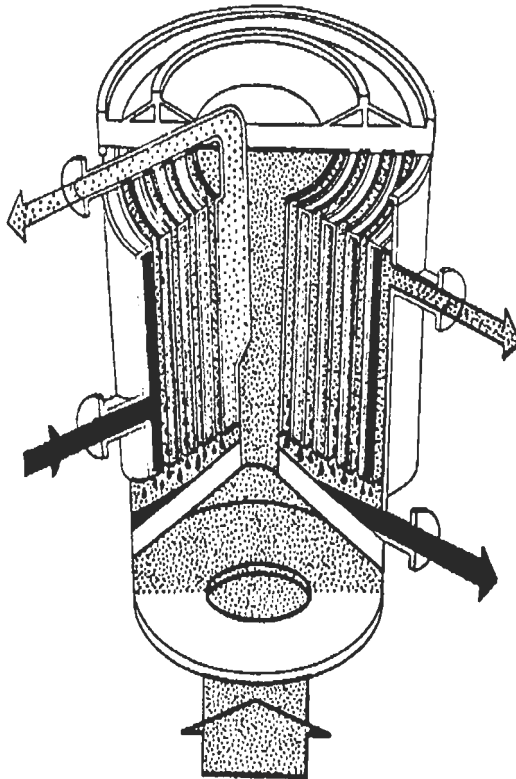
Figure 11. *Modified combination flow in a column mounted design.*

The following summarizes some of the advantages these designs have over the shell-and-tube exchanger, along with general disadvantages:

Advantages over Shell and Tube Exchangers

1. Single flow passages make it ideal for cooling or heating slurries or sludge. Slurries can be processed in the spiral-plate at velocities as low as 2 feet per second. For some sizes and design pressures, eliminating or minimizing the spacer studs enable this exchanger to handle liquids having a high content of fibrous materials.

2. Fluid distribution is good because of the single flow passage.

3. The spiral-plate exchanger generally fouls at much lower rates than the shell-and-tube exchanger because of the single flow passage and the curved flow path. If fouling does occur, the spiral-plate can be effectively cleaned chemically because of the single flow path. Because the spiral can be fabricated with identical flow passages for the two fluids, it is used for services in which the switching of fluids allows one fluid to remove the fouling deposited by the other. The maximum plate width of six feet and alignment of spacer studs permit the spiral-plate to be easily cleaned with high-pressure water or steam.

4. The spiral-plate is well suited for heating or cooling viscous fluids because its length to diameter (L/D) ratio is lower than that of straight tubes or channels. Consequently, laminar-flow heat transfer is much higher for spiral plates. When heating or cooling a viscous fluid, the spiral should be oriented with the axis horizontal. With the axis vertical, the viscous fluid stratifies and the heat transfer is reduced as much as 50 percent.

5. With both fluids flowing spirally, countercurrent flow and long passage lengths enable close temperature approaches and precise temperature control. Spiral-plates frequently can achieve heat recovery in a single unit which would require several tubular exchangers in series.

6. Spiral-plates avoid problems associated with differential thermal expansion in noncyclic services.

7. In axial flow, a large flow area affords a low pressure drop and can be of especial advantage when condensing under vacuum or when used as a thermosiphon reboiler.

8. The spiral-plate exchanger is compact: 2000 square feet of heat transfer surface can be obtained in a unit 58 inches in diameter with a 72 inch wide plate.

Disadvantages

1. The maximum design pressure is 150 psi, except for some limited sizes.

2. Repair in the field is difficult. A leak cannot be plugged as in a shell-and-tube exchanger. However, the possibility of leakage is much less in the spiral-plate because it is fabricated from plate that is generally much thicker than tube walls and stresses associated with thermal expansion are virtually eliminated. Should a spiral-plate need repairing, removing the covers exposes most of the welding of the spiral assembly. Repairs on the inner parts of the plates are complicated, however.

3. The spiral-plate exchanger is sometimes precluded from service in which thermal cycling is frequent. When used in cycling services, the mechanical design must be altered to provide for the higher stresses associated with cyclic services. Full-faced gaskets of compressed asbestos are not generally acceptable for cyclic services because the growth of the spiral plates cuts the gasket, which results in excessive fluid bypassing and, in some cases, erosion of the cover. Metal-to-metal seals are generally necessary when frequent thermal cycling is expected.

4. The spiral-plate exchanger usually should not be used when a hard deposit forms during operation, because the spacer studs prevent such deposits from being easily removed by mechanical cleaning. When, as for some pressures, spacer studs can be omitted, this limitation is not present.

PLATE-AND-FRAME EXCHANGERS

The plate-and-frame heat exchanger has emerged as a viable alternative to shell-and-tube exchangers for many applications throughout the chemical process industries. Such units are comprised of a series of plates, mounted in a frame and clamped together. Space between adjacent plates form flow channels, and the system is arranged so that hot and cold fluids enter and exit through flow channels at the four corners, as illustrated in Figure 12. Within the exchanger, an alternating gasket arrangement diverts the hot and cold fluids from each inlet into an alternating sequence of flow channels.

In this arrangement, each cell of heat transfer media is separated by a thin metal wall, allowing heat to transfer easily from one media to the other. The plate-and-frame's highly efficient countercurrent flow typically yields heat transfer coefficients three to five times greater than other types of heat exchangers. As a result, a more-compact design is possible for a given heat-exchange capacity, relative to other exchanger configurations.
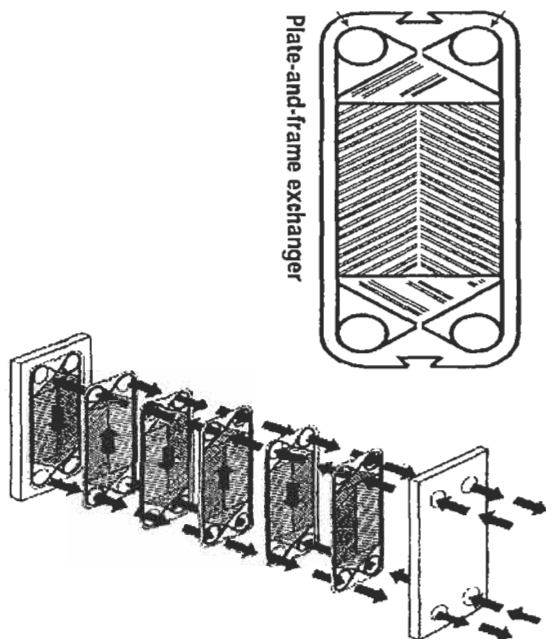
Figure 12. *Plate-and-frame exchanger.*

In this design, a corrugated chevron or herringbone pattern is pressed into each plate for several reasons. First, the pattern gives the entire exchanger strength and rigidity. It also extends the effective surface area of plates and increase turbulence in the flow channels. Together, these effects boost heat transfer. Depending on the applications, plate selection is optimized to yield the fewest total number of channel plates. Because the plates can be easily removed, service and maintenance costs are typically lower than that of shell-and-tube exchangers. Although the plate-and-frame heat exchanger can be used in almost any application, the following selection criteria must be reconciled: (a) maximum design or working pressure is limited to 300 psi; (b) temperature limits and fluids must be compatible with gasket materials (refer to Table 4); (c) plate materials must be compatible with process media; (d) the narrow passageways in the plate-and-frame can cause high pressure drops, making the exchanger incompatible with low-pressure, high volume gas applications; (e) rapid fluctuations in steam pressures and temperatures can be detrimental to gasket life. For this reason, applications that use steam favor shell-and-tube exchangers; (f) in applications where process media contain particulate matter, or when large amounts of scaling can occur, careful consideration should be given to the free channel space between adjacent plates; (g) the plate-and-frame design is best suited for applications with a large temperature cross or small temperature approach. The temperature approach is the difference between the inlet temperature of the cold

fluid and the outlet temperature of the hot fluid. Certain exchanger designs operate better at different temperature approaches. Plate-and- frame exchangers, for example, work well at a very close temperature approach, on the order of AF. For shell-and-tube exchangers, however, the lowest possible temperature approach is 10-ME. As for cleanliness of the process fluids, shell-and- tube exchangers have tube diameters that can accommodate a certain amount of particulate matter without clogging or fouling. Plate-and-frame exchangers, however, have narrow passageways, making them more susceptible to damage from precipitation or particulate fouling.

Until recently, a major limitation to the plate-and- frame exchanger was the gluing method used to attach the gaskets to the plates during construction. The glue was often applied unevenly, greatly increasing the chance of process fluid leaking through the gasket groove of the plate and either intermingling with other fluids or escaping to the atmosphere. With more modern designs, exchanger manufacturers offer a new glueless gasket system. The plate construction uses clips and studs to secure gaskets to the plates. This method eliminates irregularities in the gasket groove and results in better sealing of the plate pack. The new glueless system also cuts service and maintenance costs, since the plates can be cleaned or regasketed without removing them from the frame. However, for high-fouling applications where plates must be opened, removed and cleaned frequently, the glued gasket system may be a better choice.

Table 4. *Common Gasket Materials* [a]

Gasket Material	Max. Operating Temp., °F	Compatible Fluids
Nitrile	230	Mineral Oils, Most Aqueous Solutions, Aliphatic Hydrocarbons, Inorganics (at low concentrations and temperatures)
EPDM (Ethylene Propylene Diene Monomer Rubbers)	320	Steam, High-temperature Aqueous Solutions, Inorganic Acids and Organic Acids or bases
Viton [b]	212	Mineral Oils, Aliphatic and Aromatic Hydrocarbons, Sulfur Carbon Carbons, Trichloroethylene, Perchliroethylene

Footnotes: (a) Gaskets are also available in other materials, such as hydrogenated nitrile, neoprene, butyl rubber, hypalon, silicon rubber to meet various application requirments. (b) Viton is a Du Pont Co. trademark for a series of fluoroelastomers based on the copolymer of vinylidene fluoride and hexafluoropropylene.

Other types of plate-and-frame heat exchangers are double-wall-plate exchangers, welded-plate exchangers, wide-gap-plate exchangers, and brazed-plate exchangers. Each type is briefly described below.

Double-Wall-Plate Exchangers

Another recent advance in plate-and-frame design is the double-wall plate heat exchanger, which offers greater protection against gasket failure. In traditional plate-and-frame exchangers, the process fluids are contained by gaskets and thin, metal plates. In double-wall exchangers, two standard plates are welded together at the port holes to form one assembly, with an air space between the plates. Any leaking fluid is thus allowed to collect in this interstitial space, instead of entering an adjacent fluid passageway and contaminating the other process stream. Typical applications include domestic water heaters, hydraulic oil cooling, any service where cross contamination of process fluids cannot be tolerated.

Welded-Plate Exchangers

In this design, the field gasket that normally contains the process fluid is replaced by a welded joint. When plates are welded together at the periphery, leakage to the atmosphere is prevented, so this design is suitable for hazardous or aggressive fluids. The welded plates form a closed compartment or "cassette." Similar to gasketed designs, alternating flow channels are created to divert the flow of hot and cold fluids into adjacent channels. Aggressive fluids pass from one cassette to the next through an elastomer or Teflon ring gasket, while non-aggressive fluids are contained by standard elastomer gaskets. The use of welded joints can reduce total gasket area by 90% on the aggressive-fluid side. Typical applications include exchangers handling vaporizing and condensing refrigerants, corrosive solvents and amine solutions.

Wide-Gap-Plate Exchangers

Compared with traditional plate-and-frame exchangers, this design relies on a more loosely corrugated chevron pattern, which provides exceptional resistance to clogging. The plates are designed with few, if any, contact points between adjacent plates to trap fibers or solids. Some styles of this exchanger use wide-gap plates on the process side and conventional chevron patterns on the coolant

side, to enhance heat transfer. Typical applications include exchangers handling white water in pulp-and-paper operations, and various slurries.

Brazed-Plate Exchangers

This represents one of the latest technological advance in plate heat exchangers - the elastomer gaskets found in most plate exchangers are replaced with a brazed joint, which greatly reduces the possibility of leakage. The heat transfer plates, which are only available in Type 316 stainless steel, are brazed together using either a copper or nickel brazing material. These exchangers are built to manufacturers standards and are not often offered as stock items. The brazed-plate exchangers are typically ASME rated to 450 psi. Temperature ratings vary from 375 °F for copper brazing to 500 °F for nickel brazing. As with other plate-and-frame exchangers, high heat transfer rates translate to compact designs. Typical applications include units that vaporize and condense refrigerants, process applications requiring high alloys, heat-recovery applications, brine exchangers, applications involving liquid ammonia, chlorine solutions, alcohols or acids.

HEAT EXCHANGER TUBE RUPTURE

A tube rupture in a shell and tube heat exchanger is a safety concern when there is a significant pressure difference between the shell and tube sides, particularly when the low pressure side is liquid filled. In the event of a tube rupture in such an exchanger, the high pressure fluid will flow through the ruptured tube and can quickly overpressure the low pressure side of the exchanger. A typical refinery example is in a hydroprocessing unit where there may be reactor effluent on the high pressure side and cooling water or liquid hydrocarbon on the low pressure side. Using dynamic simulation to include inertia of the fluid and expansion of the exchanger shell, pressure spikes associated with different exchanger designs can be determined. Dynamic simulation becomes a valuable tool in determining the relief device size and location or in setting the heat exchanger mechanical design that minimizes these effects. Dynamic simulation can also save an expensive replacement during a revamp if an existing heat exchanger design can be proven adequate for the tube rupture case even though it may not meet the two-thirds rule. Dynamic simulation is a process engineering design tool that predicts how a process and its controls respond to various upsets as a function of time. Dynamic simulation can be used to evaluate equipment configurations and control schemes and to determine the reliability and safety of a design before capital is committed to the project. For grassroots and revamp projects, dynamic

simulation can be used to accurately assess transient conditions that determine process design temperatures and pressures. In many cases, unnecessary capital expenditures can be avoided using dynamic simulation.

Dynamic simulation during process design leads to benefits during plant startup. Expensive field changes which impact schedule can often be minimized if the equipment and control system is validated using dynamic simulation. Startup and shutdown sequences can be tested using dynamic simulation. Dynamic simulation also provides controller tuning parameters for use during startup. In many cases, accurate controller settings can prevent expensive shutdowns and accelerate plant startup.

Dynamic simulation models used for process design are not based on transfer functions as normally found in operator training simulators, but on fundamental engineering principles and actual physical equations governing the process. When used for process design, dynamic simulation models include: (a) equipment models that include mass and energy inventory from differential balances; (b) rigorous thermodynamics based on property correlations, equations of state, and steam tables; actual piping, valve, distillation tray, and equipment hydraulics for incompressible, compressible, and critical flow; (c) detailed controller models to duplicate modern distributed control systems (DCS).These models are so detailed that the results can influence engineering design decisions and ensure a realistic prediction of the process and the control system's interaction to assess control system stability.

When dynamic simulation is used for process equipment and process safety design, it is necessary to ensure the model's assumptions are conservative. For example, if dynamic simulation is used to calculate the pressure rise in a heat exchanger after a tube rupture, the highest calculated pressure may be used as the design pressure. If all the assumptions are conservative, the actual heat exchanger pressure will not exceed the design pressure during a tube rupture. Despite this conservative approach, equipment design conditions calculated by dynamic simulation are often much less severe than the conditions determined by conventional calculation methods. This often leads to considerable cost savings.

Dynamic simulation software should support the addition of user-written code for specialized equipment and control system models. For example, an unusual fractionator tray design or a correlation for an off-design heat transfer coefficient may have to be programmed into a user-written model. Dynamic simulation of "first-of-a-kind" plants often requires developing a dynamic model for a new equipment item. A control system vendor's DCS algorithm may also need to be programmed into a custom PID controller model. Users may need to add their own fluid property systems to increase computational efficiency and handle unusual systems. "Black box" models are too restrictive to provide realistic models for most dynamic simulation problems.

During process design, the greatest opportunity to benefit from dynamic simulation is after adequate design information is available to develop the model,

but early enough so simulation results can be incorporated into the design. Dynamic simulation requires detailed design information such as system volumes, control valve C_vs, and heat-transfer surface areas. However, once this design data is available, often little time exists to complete the dynamic model before the process design is fixed and equipment must be purchased. For this reason, dynamic simulation is performed under very demanding time requirements. It is essential that the modeling goals be clearly understood and that the model contains only those features that are required to answer key design questions.

Effective simulations should be developed and executed by process engineers who are aware of simulation objectives, capabilities, and limitations and also the needs of design engineers responsible for detailed system design. The dynamic simulation should be performed at the same physical location as the process design work to provide better design data access and the daily opportunity to discuss problems and solutions with design engineers. This synergy produces a model that addresses key design issues and provides results that can be incorporated into the design early in the project's schedule.

Typical dynamic simulation applications include hydrocracker/hydrotreater depressuring analysis, distillation/fractionation column relief load reduction, heat exchanger tube rupture transient analysis, refinery steam production and distribution system control analysis, compressor surge control analysis, vacuum unit transfer line designs at steady-state, cryogenic depressuring studies, and distillation/fractionation column control analysis. In addition to these typical applications, dynamic simulation can be applied to "first-of-a-kind" plants. These plants are especially prone to unforeseen process and control system interactions during transient conditions. Dynamic simulation can highlight these problems early in the design phase to avoid costly post-startup modifications.

A tube rupture transient analysis is a dynamic simulation of a shell-and-tube heat exchanger where there is a significant difference between the shellside and tubeside design pressures, particularly when the low-pressure side is liquid-filled. When a tube ruptures, high-pressure fluid flows through the ruptured tube and can quickly overpressure the exchanger's low-pressure side. Many heat exchangers in refinery hydroprocessing units have 100 to 200 barg (1500 to 3000 psig) reactor effluent or recycle gas on the tubeside with 3.5 barg (50 psig) cooling water on the shellside. For these heat exchangers such as these, API recommends dynamic simulation of the tube rupture. It is required in many refiners' design practices.

If a tube breaks, pressure on the exchanger low-pressure side can spike to a level that exceeds the pressure predicted by a steady-state analysis. This spike is due to pressure buildup before the fluid accelerates out of the shell and/or before the relief device fully opens.

Dynamic simulation models include fluid inertia and compressibility and exchanger shell expansion to determine the pressure spikes associated with

different exchanger designs. Therefore, dynamic simulation becomes a valuable tool in determining the size and location of relief devices, or the heat exchanger mechanical design required to prevent overpressuring during a tube rupture. In many cases, dynamic simulation can save an expensive heat exchanger replacement during a revamp if the existing exchanger can be rerated for the tube rupture case.

The API recommended practice (per API RP-521), notes that tube failure should be considered as a viable relief scenario. A tube rupture transient analysis is recommended for heat exchangers where the low pressure side is not designed for two-thirds of the high pressure side design pressure or where the high pressure side operating pressure is greater than 69 barg (1000 psig). Heat exchangers whose the low-pressure side is designed for two-thirds of the high-pressure side do not require tube rupture analysis unless the operating pressure is above 69 barg. The underlying assumption for this practice is that an exchanger designed for two-thirds of the high pressure side can be blocked in after a tube rupture and the pressure will not exceed the exchanger's hydrotest pressure (vessels and heat exchangers are hydrotested at a minimum of 150% of the design pressure).
The following should be considered with the two-thirds rule:

- A tube rupture transient analysis is recommended if the operating high-pressure side is greater than 1000 psig (69 barg), even if the exchanger is designed for the two-thirds rule.
- If a heat exchanger designed for the two-thirds rule is blocked in after a tube rupture, pressure on the low-pressure side can rise to that of the high-pressure fluid and still not exceed the hydrotest pressure of the low-pressure side. However, inlet and outlet piping up to and including the isolation valves must also be designed for the two-thirds rule if the exchanger will be blocked in.
- Designing heat exchangers for the two-thirds rule became common in the early 1980s. Before then, heat exchangers were rarely designed for tube ruptures. Consequently, many refinery units over 15 years old contain heat exchangers and piping that do not satisfy the two-thirds rule. Many refiners are now systematically evaluating these older exchangers for tube rupture due to OSHA-mandated HAZOP analysis.

A heat exchanger tube rupture occurs in two stages: Stage 1: The tube breaks and high-pressure fluid displaces the low-pressure fluid out the exchanger's low-pressure side. High-pressure fluid does not flow through the connecting low-pressure piping. Stage 2: High-pressure fluid flows out through the low-pressure piping. Stage 2 consists of fully developed, often two-phase flow of low- and high-pressure fluid from the heat exchanger through downstream piping.
Stage 1 may last less than one second to several seconds. It is characterized by a very fast transient and a pressure spike immediately after the tube rupture. After the low-pressure side fills with high-pressure fluid, the transition to stage 2

occurs and high-pressure fluid begins to flow downstream. Stage 2 lasts indefinitely high-pressure fluid is discontinued. Exchanger pressure usually decreases in stage 2 because the outlet fluid density is normally substantially lower. The dynamic simulation usually only attempts to model stage 1. The transition to stage 2, and stage 2 itself, are usually not simulated in the transient analysis.

Design considerations should be examined by process design engineers when designing heat exchangers for stage 1 tube rupture transient effects, which includes the following:

- For revamps, exchangers with the low-pressure fluid on the tubeside can sometimes be re-rated for the two-thirds rule without any equipment redesign. However, the inlet and outlet piping up to and including the isolation valves may also need to be re-rated or redesigned for the two-thirds rule.

- The design pressure of a heat exchanger and other equipment and piping may be exceeded during a tube rupture of an adjacent exchanger if it operates at a high pressure.

- When low-pressure fluid is on the shellside, it may be possible to protect the exchanger with a relief device on the inlet or outlet piping. However, when the low-pressure fluid is a tubeside liquid, preventing a significant pressure spike in the heat exchanger channel volumes is very difficult. A relief device on the inlet and outlet piping may not protect the exchanger because the relief device will not open before the channels' design pressure is exceeded.

- A vapor pocket on the exchanger's low-pressure side can create a cushion that may greatly diminish the pressure transient's intensity. A transient analysis may not be required if sufficient low-pressure side vapor exists (although tube rupture should still be considered as a viable relief scenario). However, if the low-pressure fluid is liquid from a separator that has a small amount of vapor from flashing across a level control valve, the vapor pocket may collapse after the pressure has exceeded the fluid's bubble point. The bubble point will be at the separator pressure. Transient analysis will predict a gradually increasing pressure until the pressure reaches the bubble point. Then, the pressure will increase rapidly. For this case, a transient analysis should be considered.

In a tube rupture model (refer to API RP-521), the low-pressure fluid is on the shellside. An instantaneous rupture of a single tube is assumed to occur at the tube sheet. An API model calculates flow through the tube sheet orifice and through the long tube from the opposite tube channel. If the low-pressure side is on the tubeside, then the model must calculate the flow through the short tube break into one tube channel, and through the long tube length to the opposite tube

channel. The model must be able to calculate either critical or subcritical flow and the transition from one to the other.

High-pressure fluid flows into the low-pressure shell (or tube channel if the low-pressure fluid is on the tubeside). The low-pressure volume is represented by differential equations that determine the accumulation of high-pressure fluid within the shell or tube channel. The model determines the pressure inside the shell (or tube channel) based on the accumulation of high-pressure fluid and remaining low pressure fluid. The surrounding low-pressure system model simulates the flow/pressure relationship in the same manner used in water hammer analysis. Low-pressure fluid accumulation, fluid compressibility and pipe expansion are represented by pipe segment symbols. If a relief valve is present, the model must include the spring force and the disk mass inertia.

To summarize, a tube rupture in a shell and tube heat exchanger is a safety concern when there is a significant pressure difference between the shell and tube sides, particularly when the low pressure side is liquid-filled. In the event of a tube rupture in such an exchanger, the high pressure fluid will flow through the ruptured tube and can quickly overpressure the low pressure side of the exchanger. API's Guide for Pressure-Relieving and Depressuring Systems (RP-521, Fourth Edition, March 1997) states the following guidelines:

- Pressure relief is not required when the heat exchanger, including upstream and downstream piping and equipment, is designed for two-thirds of the high pressure side design pressure (section 3.18.2).
- Transient Analysis (dynamic simulation) is recommended where there is a wide difference in design pressure between the two exchanger sides, especially where the low-pressure side is liquid-filled (section 3.18.3).

The first guideline is often referred to as the "two-thirds rule." The basis of this rule is that if the low pressure side is designed for two-thirds of the high pressure side design pressure, the exchanger hydrotest pressure will not be exceeded due to a tube rupture.

The 1997 edition of the API RP 521 extends the two-thirds rule to include the upstream and downstream system. At a minimum, the inlet and outlet piping up to and including isolation valves must be designed for the two-thirds rule to be able to block in the exchanger. If the upstream and downstream equipment is not designed for the two-thirds rule, relief devices may be required on both the inlet and outlet piping to protect the piping and adjacent equipment.

API RP-521 recommends transient analysis for exchangers with wide difference in design pressure (such as cases where the two-thirds rule was not applied) because the pressure in the low pressure side of the exchanger can spike to a level that exceeds the pressure predicted by a steady state analysis when it is liquid-filled. This pressure spike is due to pressure buildup before the liquid is accelerated out of the low pressure side and/or before the relief device opens fully. API RP-521 recommends that the basis for the tube rupture be a sharp

break of a single tube. Refinery experience has shown that tube failures can range from slow leaks to cases where several tubes have ruptured simultaneously.

Per the 1997 API RP-521, tube rupture no longer must be considered for exchangers designed for the two-thirds rule when the high-pressure side design pressure is over 1000 psig (69 barg). In the previous API RP-521 edition, tube rupture should be considered for all exchangers where the high pressure side design pressure was over 1000 psig even if the exchanger was designed for the two-thirds rule. Many large refiners have design practices (e.g. Texaco GEMS, and Exxon) which closely follow the API guidelines and require transient analysis for specific tube rupture cases.

Fluor Daniel has the ability to perform a heat exchanger tube rupture transient analysis consistent with the method referred to in RP-521 ("Model to Predict Transient Consequences of a Heat Exchanger Tube Rupture," by Sumaria et al.). This methodology accounts for effects such as the inertia of the low-pressure liquid, the compressibility of the liquid, the expansion of the exchanger shell or tube channels, and the relief valve dynamics. Dynamic simulation can be used to meet the following objectives:

- Determine the heat exchanger design conditions required to prevent overpressuring during tube rupture. Many refineries contain units with heat exchangers that were not designed for the two-thirds rule. These exchangers are often targeted for replacement after a HAZOP (Hazard Operability) review or during a revamp. In these circumstances, dynamic simulation can often save an expensive heat exchanger replacement if the existing exchanger can be rerated for the tube rupture case.
- Determine the size and location of relief devices required to protect an exchanger from overpressure during a tube rupture.
- Investigate the effect of the pressure surge on adjacent equipment per the 1997 edition of API RP-521. The design pressure of adjacent equipment and piping may be exceeded during a tube rupture. This is of special concern in cooling water networks. Dynamic simulation can assess the impact of a tube rupture on adjacent equipment and identify corrective measures.

Fluor Daniel reported the following tube rupture analysis for a hydrotreater feed/effluent heat exchanger for a Southern California refinery. The analysis was initiated as a unit revamp resulted in the exchanger's low pressure side being completely liquid filled. In the original design, the low pressure side of the exchanger was filled with a mix of vapor and liquid. A tube rupture could be much more severe for the revamp design. The tube side operating pressure for the original and revamp design was 100 barg (1500 psig). The shell side operating pressures was approximately 10 barg (150 psig) for the original design and 4 barg (60 psig) for the revamp case. In the original design, a tube rupture transient analysis set the design pressure of the shell and adjacent equipment and

piping at approximately 20 barg (300 psig). The dynamic simulation showed that the pressure in the exchanger shell reached a peak of 45 barg (650 psig) 0.12 seconds after the rupture occurred. The dynamic simulation also predicted a peak pressure of 43 barg (625 psig) in a filter downstream of this exchanger. A mechanical analysis of the exchanger shell indicated that the exchanger could be rerated with a design pressure equal to the peak pressure predicted by the transient analysis if the corrosion allowance was reduced below the original design value. All of the piping was designed for 300 pound line class and did not require modification. Therefore, as a result of dynamic simulation, the existing exchanger and piping were able to be saved. However, a mechanical analysis of the filter indicated that the filter housing would have to be replaced to withstand the pressure surge.

CONDENSERS

Condensation is the process of reduction of matter into a denser form, as in the liquefaction of vapor or steam. Condensation is the result of the reduction of temperature by the removal of the latent heat of evaporation. The removal of heat shrinks the volume of the vapor and decreases the velocity of, and the distance between, molecules. The process can also be thought of as a reaction involving the union of atoms in molecules. The process often leads to the elimination of a simple molecule to form a new and more complex compound.

Condensation heat transfer is a vital process in the process and power generation industries. The existing modes of condensation are **filmwise** and **dropwise**. Filmwise is currently used by industry, while dropwise is an alternative which is under development because it offers attractive higher rates of heat transfer by preventing the build up of the insulating liquid layer found in filmwise condensation. All but a few precious metals will in an untreated state tend to condense filmwise: this is why industrial condensers operate filmwise. The type of condensation behavior which a metal displays is related to it's surface energy. Materials with a high surface energy condense filmwise while those with a low surface energy condense dropwise. With suitable promoters or surface treatments, most metals, including those with high surface energies, can promote dropwise condensation.

Non-azeotropic mixtures have been utilized in refrigeration systems for several direct and indirect advantages like, enhanced coefficient of performance, lower power consumption, reduced thermal irreversibility, increased chemical stability, improved oil miscibility, varying condensation temperatures and variable capacity refrigeration systems. All these merits offer rich prospects for the use of mixed component working fluids in heat pumps, power cycles and refrigeration systems. In the mid 1980s, a new thermodynamic power cycle using a multicomponent

working fluid as ammonia-water with a different composition in the boiler and condenser was proposed (known as the Kalina cycle). The use of a non-azeotropic mixture decreases the loss of availability in a heat recovery boiler when the heat source is a sensible heat source, and in a condenser when the temperature decreases with heat exchange. Most heat input to a plant's working fluid is from variable temperature heat sources. Due to its variable boiling temperature, the temperature rise in an ammonia-water mixture in a counterflow heat-exchanger closely follows the straight line temperature drop of the heat source. Although this is an advantage over the conventional single component Rankine cycle, given equal condenser cooling temperatures, the ammonia-water mixture will have a significantly higher pressure and temperature than the steam at the condensing turbine outlet. The higher pressure is a result of ammonia being more volatile than water. The higher temperature is a result of variable condensing temperature of the ammonia-water mixture. In the Rankine cycle much of the heat (almost 65%) from a turbine exhaust cannot be recuperated because there is no temperature difference between the steam at the turbine exhaust and the water at the condenser outlet. However, in the Kalina cycle, much of this latent heat can be extracted due the higher temperature of the turbine exhaust over the ambient coolant temperature.

The need for effectively extracting latent heat from the turbine exhaust in the Kalina cycle is the motivation behind numerous studies of convective condensation of non-azeotropic vapor mixtures. In condensers operating with pure vapors, the vapor pressure generally remains constant during the process of phase change. Therefore, it implies that the temperature difference between the vapor and the coolant increases along the direction of vapor flow in a counterflow type of heat exchanger. Thus, a situation is created in which the available excess energy is maximum at the exit of the condensate and minimum at the entrance of the entrance of the vapor. As a result, all the available energy is not utilized in pure vapor condensation. The utilization of availability can be enhanced by maintaining a constant temperature difference between the vapor and coolant, all along the heat exchanger. This can be achieved by using a certain non-azeotropic vapor mixture which can maintain a constant temperature difference due to its variable boiling temperature characteristics. The introduction of another condensable vapor, may alter the composition of the vapor and decrease the heat and mass transport in the condenser. Furthermore, the orientation of the condenser can affect the flow regime in the condenser, and hence alter the performance of the condenser.

Although condensers likely warrant a separate chapter, conventional equipment often used alongside with heat exchanger are described here. In surface and contact condensers, the vapors can be condensed either by increasing pressure or extracting heat. In practice, condensers operate through removal of heat from the vapor. Condensers differ principally in the means of cooling. In surface

condensers, the coolant does not contact the vapors or condensate. In contact condensers, coolant, vapors, and condensate are intimately mixed. Most surface condensers are of the tube and shell type shown in Figure 13.

Water flows inside the tubes, and vapors condense on the shell side. Cooling water is normally chilled, as in a cooling tower, and reused. Air-cooled surface condensers and some water-cooled units condense inside the tubes. Air-cooled condensers are usually constructed with extended surface fins.

Most vapors condense inside tubes cooled by a falling curtain of water. The water is cooled by air circulated through the tube bundle. The bundles can be mounted directly in a cooling tower or submerged in water.

Contact condensers employ liquid coolants, usually water, which come in direct contact with condensing vapors. These devices are relatively uncomplicated, with typical configurations illustrated in Figure 14. Some contact condensers are simple spray chambers, usually with baffles to ensure adequate contact. Others, incorporate high-velocity jets designed to produce a vacuum.

In comparison to surface condensers, contact condensers are more flexible, are simpler, and considerably less expensive to install. On the other hand, surface condensers require far less water and produce 10 to 20 times less condensate than contact type condensers.
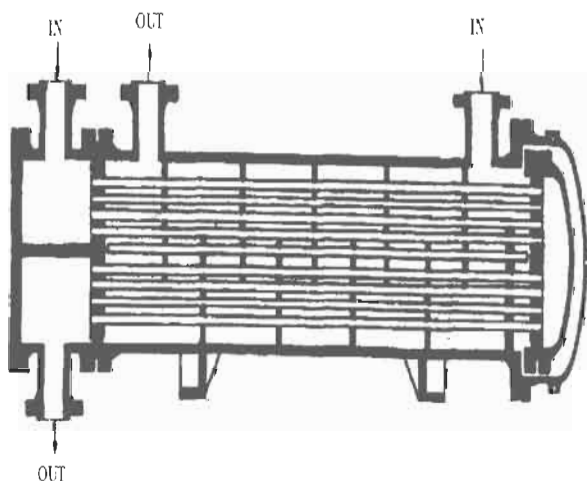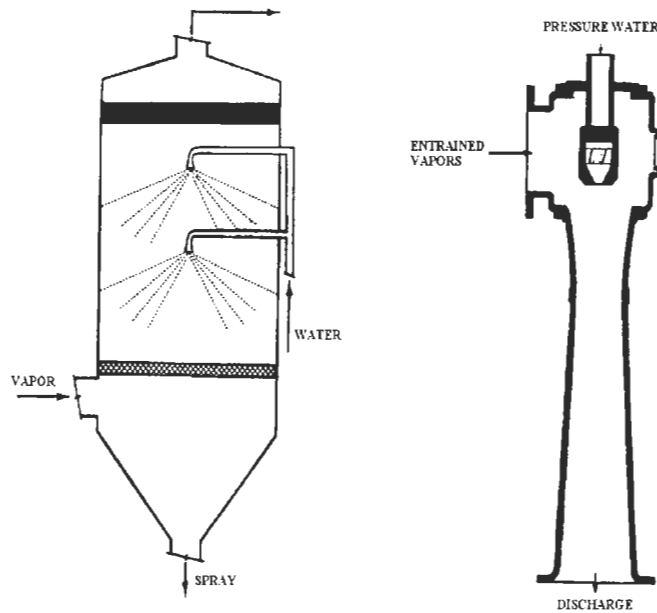

Figure 13. *Shell and tube condenser.*

Figure 14. *Common condenser configurations.*

Condensate from contact units cannot be reused and may constitute a waste disposal problem. Surface condensers can be used to recover valuable condensate. Surface condensers must be equipped with more auxiliary equipment and generally require a greater degree of maintenance.

In general, subcooling requirements are more stringent for surface units than for contact condensers, where dilution is much greater. Nevertheless, many surface condenser designs do not permit adequate condensate cooling. In the typical water-cooled, horizontal, tube-and-shell condenser (Figure 15), the shell side temperature is the same throughout the vessel. Vapors condense, and condensate is removed at the condensation temperature, which is governed by pressure. In a horizontal-tube unit of this type, condensate temperature can be lowered by: (1) reducing the pressure on the shell side, (2) adding a separate subcooler, or (3) using the lower tubes for subcooling. Vertical-tube condensers provide some degree of subcooling even with condensation on the shell side. With condensation inside the tubes, subcooling occurs in much the same manner whether tubes are arranged vertically or horizontally. With inside-the-tube condensation, both condensate and uncondensed vapors pass through the full tube length. A separate hot well is usually provided to separate gases before the condensate is discharged. Water requirements for contact condensers can be calculated directly from the condensation rate, by assuming equilibrium conditions.
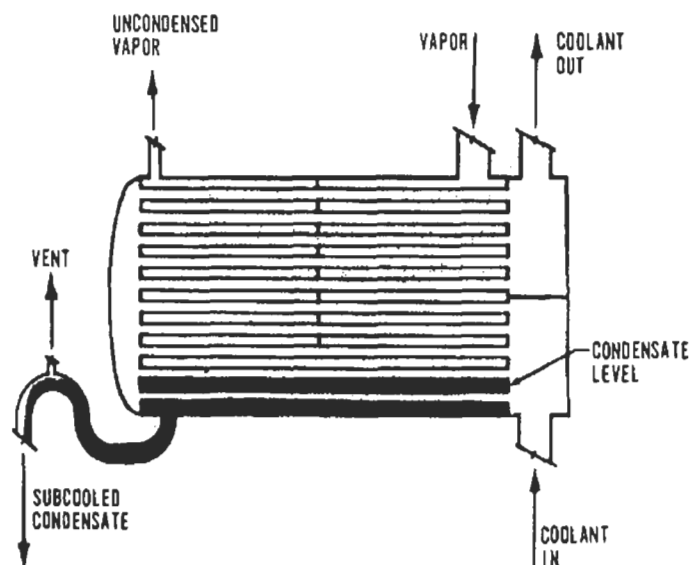
Figure 15. *Subcooling arrangement in a horizontal tube and shell condenser.*

The cooling water (or other medium) must absorb enough heat to balance the heat of vaporization and condensate subcooling. Piping and hot wells must be sized based upon the maximum condenser requirement. The following example illustrates the method of calculating the quantity of cooling water for a specific service.

Example: Exhaust vapors from a process operation contain 95 percent steam at 200 °F at 11.5 psia. The maximum evaporation rate in the cooker is 2,000 lb per hour. Steam is to be condensed at 200 °F and cooled to 140 °F in a contact condenser. A vacuum pump removes uncondensable vapors at the condenser and maintains a slight vacuum on the cooker. Determine the volume of 60 °F fresh water required and the resultant condensate volume. The solution to this problem is as follows:

Condensation = 2,000 × 977.9 Btu/hr = <u>1,960,000 Btu/hr</u>

Subcooling = 2,000 × (200-140) Btu/hr = <u>120,000 Btu/hr</u>

Cooling load = <u>2,080,000 Btu/hr</u>

Water requirement = 2,080,000 Btu/hr ÷ (140-60) Btu/lb

    = 26,000 lb/hr = <u>51.4 gpm</u>

Total condensate = 51.4 + 2,000 lb/hr ÷ (60 x 8.33 lb/gal)

    = <u>55.4 gpm</u>

As noted earlier, condensation occurs through two distinct physical mechanisms, namely drop-wise and filmwise condensation. When a saturated pure vapor comes in contact with a sufficiently cold horizontal surface, the vapor condenses and forms liquid droplets on the surface. These droplets fall from the surface, leaving bare metal exposed on which successive condensate drops may form. This is known as dropwise condensation. Normally, a film occurs and coats the condensing surface. Additional vapors must then condense on this film rather than on the bare metal surface. This is called film-wise condensation and occurs in most condensation processes. Heat transfer coefficients are one-fourth to one-eighth the transfer units associated with dropwise condensation. Steam is the only pure vapor known to condense in a dropwise manner. Dropwise condensation has been found to take place at various times when a mixture of vapors and gases is present. Some degree of dropwise condensation may possibly be attained by using certain promoters. Promoters such as oleic acid on nickel or chrome plate, and benzyl mercaptan on copper or brass become absorbed on the surface as a very thin layer to prevent the metal surface from being wetted by any condensate. Steel and aluminum surfaces are difficult to treat to acquire dropwise condensation. Use of these promoters increases the heat transfer coefficient to 6 to 10 times the amount of film-wise coefficients. Nearly all condenser design calculations are based on heat transfer that is affected by an overall transfer coefficient, temperatures, and surface area. A mathematical solution to the problem is usually achieved by the expression:

$$Q = UAT_m$$

where: Q = heat transferred, Btu/hr

   U = overall coefficient, Btu/hr per ft per °F

   A = heat transfer area, ft^2

   T_m = mean temperature difference, °F

Condenser design is often more difficult than indicated by the foregoing expression, and a simplified or general overall heat transfer coefficient is not used. This is especially true when a vapor is condensed in presence of a noncondensable gas. Nusselt relations were developed for streamlined flow of all vapor entering vertical- or horizontal-tube exchangers. These equations account for the variation of the film thickness (thinnest at top of the tube and tube bundle of vertical and horizontal exchangers) by expressing the vapor side mean heat transfer coefficient in terms of condensate loading. In instances of streamlined flow of condensate, the heat-transfer coefficient has been established as inversely proportional to film thickness. Observations have, however, shown a decrease to a certain point, and then a reverse effect when the coefficient increased. This

reversal occurs at a Reynolds number of approximately 1,600, indicating that turbulence in liquid film increases the heat transfer coefficient.

A temperature profile of vapor condensing in the presence of a noncondensable gas on a tube wall, as shown in Figure 16 indicates the resistance to heat flow. Heat is transferred in two ways from the vapor to the interface. The sensible heat is removed in cooling the vapor from t_v to t_c at the convection gas cooling rate. The latent heat is removed only after the condensable vapor has been able to diffuse through the noncondensable part to reach the tube wall. This means the latent heat transfer is governed by mass transfer laws.

Some general facts about condenser operations should be noted:

- Any saturated vapor can be condensed by a direct spray of cold water under correct temperature and pressure. If sufficient contact is provided, coolant and vapor will reach an equilibrium temperature. The condensate created by the water should not be objectionable in its liquid form.

- Pure vapor or substantially pure vapor can be considered condensed isothermally, and during the condensate range the latent heat of condensation is uniform.
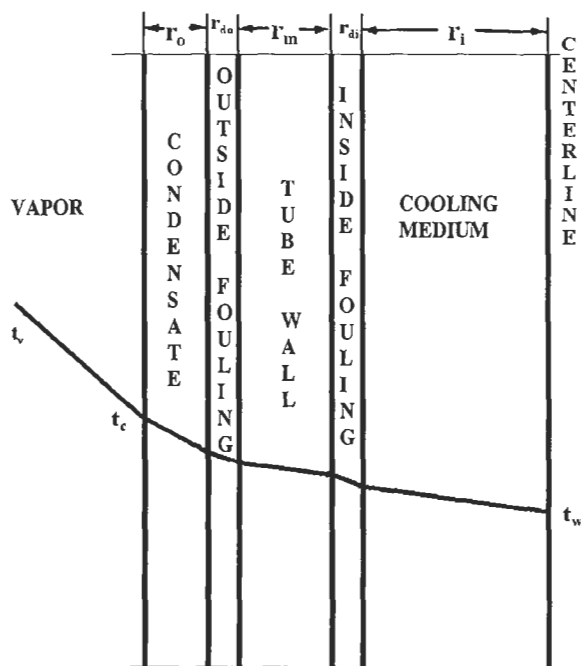

Figure 16. *Temperature profile showing effect of vapor condensation on a tube wall in the presence of a noncondensable gas.*

- If the temperature range of a mixture does not exceed 10° to 20°F, condensation of this mixture may be treated as a pure component.

- In condensation of streams consisting primarily of steam, the condenser size ranges from 10, 000 to 60,000 square feet per shell (bundle), the tubes averaging 26 feet long.

- In water-cooled tube-and-shell condensers with shell side condensation, overall heat transfer coefficients for essentially pure steam range from 200 to 800 Btu per hour per square foot per °F.

- With tube side condensation, coefficients are generally lower than for comparable shell side condensers. This phenomenon is attributed to: (1) lower coolant velocities outside the tubes than are possible with tube side cooling, and (2) increased film thicknesses, namely, film resistance inside the tubes.

- Noncondensable gases at condenser temperature blanket the condenser surface and reduce the condenser capacity.

- Condensation reduces the volume of the vapor present and can be assumed to occur at a constant pressure drop.

- A balanced pressure drop maybe assumed in the horizontal condenser where partial condensation is occurring.

- Within low-pressure operating ranges, the slight pressure loss due to friction in vapor pipes may mean an appreciable loss of total available temperature difference.

- Low-density steam under vacuum conditions can cause a linear velocity to be higher than is allowable with steam lines.

- Vapors should travel across the bundle as fast as possible.

- Air or inerts can cause up to 50 percent reduction in condensation coefficients.

- Sources of air or inerts include: dissolved gas in the cooling water in case of jet condensers, entrainment with steam, entrainment with vapor, leaks, and noncondensable gases.

- In vertical-tube condensers, 60 percent of the condensation occurs in the upper half.

- Horizontal position of a condenser distributes the vapor better and permits easier removal of the condensate.

- In the horizontal condenser, it is necessary to prevent cooled condensate from forming liquid pools and impeding the flow of vapors.

- Selection of which material should pass through tubes cannot be decided by fixed rules, because of factors at a variance with one another. When corrosive condensate is encountered, condensation within the tubes rather than the shell is usually desirable.

The following is a partial list of common applications for condensers: In petroleum and petrochemical applications - manufacture of detergents; alkylation unit accumulator vents; manufacture of insecticides; amine stripper units; manufacture of latex; butadiene accumulator vents; manufacture of nitric acid; ketone accumulator vents; manufacture of phthalic anhydride; lube oil re-refining; resin reactors; polyethylene gas preparation accumulators; soil conditioner formulators; residium stripper unit accumulator vents; solvent recovery; storage equipment; thinning tanks;styrene-processing units; aluminum fluxing; toluene recovery accumulator vents; asphalt manufacturing. In chemicals manufacturing, applications include - degreasers; manufacture and storage of ammonia; dry cleaning units; manufacture of copper naphthenates; esterfication processes; chlorine solution preparation; vitamin formulation; manufacture of ethylene dibromide; rubber finishing operations.

STEAM-DRIVEN ABSORPTION COOLING

Concerns about energy efficiency and the use of chlorofluorocarbons have led to the greater use of absorption machines for cooling applications. These machines, which reclaim condenser or exhaust-gas heat, are used in advanced cogeneration systems. Absorption machines are increasingly used for space cooling for two main reasons: First, the machines, which use a lithium-bromide brine as working fluid, use water as a refrigerant, which is environmentally safer than other refrigerants. Second, they are driven by thermal energy, which reduces peak demand for electric power.

Absorption cooling can be activated by low-temperature energy (such as low-pressure steam, geothermal, or solar) or by high-temperature energy, (such as a gas flame). A steam-driven absorption system is equivalent to a power plant and vapor-compression cooling cycle because it receives thermal energy and rejects heat to the environment while cooling. The seasonal coefficient of performance (COP) of a good gas-fired double-effect absorption machine is comparable to that of a combination of a power plant and a good centrifugal compressor and superior to that provided by a low-efficiency compressor.

Conventional refrigeration is based on the evaporation of a refrigerant. The evaporator produces cooling, the compressor generates high-pressure energy, and the condenser rejects heat to the environment. An absorption machine differs from a vapor compressor only in the way the refrigerant is compressed from the evaporator to the condenser.

In the compression machine, the refrigerant pressure is raised by an input of mechanical energy, which drives a compressor. In the absorption machine, the refrigerant is absorbed in the absorber at its evaporator pressure by an aqueous solution of lithium bromide. The liquid solution is then pumped to the generator via a recuperator. Heat input in the generator drives the refrigerant off the solution at the condenser pressure. Heat is rejected in the absorber and condenser. In comparison to the compressor, the pump requires a small amount of mechanical energy. Single-effect cycle COPs fall in the 0.5 to 0.7 range.

When the separation of refrigerant is done via a double-effect distillation, the cycle is called a double effect. The double-effect cycle requires higher temperatures than the single-effect cycle but returns a higher COP, ranging from 1 to 1.3, depending on the machine. Triple-effect and other advanced cycles under development promise still higher performance. Because single-effect machines require low-temperature energy (about 104 °C) to produce cooling, they can use low-grade reject energy from other cycles. Absorption machines, which use rejected heat from a turbine that drives a generator or a compressor, are increasingly being used in cogeneration applications. In the most advanced cogeneration schemes in use today, high COPs (of about 3.5) are possible by cascading thermal energy from Brayton to Rankine cycles to absorption machines, with both cycles driving compressors.

CLOSURE

This chapter has only provided the most basic overview of heat exchanger equipment. For most applications and heat-exchanger types, there are a multitude of choices and options to select from. Regardless of the application, the ultimate focus of the equipment is on clean, efficient heat recovery. Given the extent of investments on the part of the CPI in heat transfer equipment, and incentives for energy conservation through heat recovery operations, heat exchanger equipment will continue to be among the most critical components in many manufacturing processes. The following are suggested references for obtaining more detailed information.

NOMENCLATURE

A   area

a   heat transfer parameter

C_p heat capacity

D, d   diameter or size

e   efficiency

h   local heat transfer coefficient

k   Boltzmann constant

l   length

m   mass

n   rotational speed

Nu   Nusselt number

P   Pressure

P_c price or cost index

Pr   Prandlt number

Re   Reynolds number

t   temperature

T   absolute temperature

q   heat transfer quantity

U   overall heat transfer coefficient

v   velocity

x   thickness

Greek Symbols

α coefficient of expansion

ρ density

Γ temperature parameter in log-mean definition

Θ temperature parameter in log-mean definition

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Chapter 2
EVAPORATIVE
COOLING EQUIPMENT

INTRODUCTION

This chapter discusses an important piece of equipment used throughout many industry sectors, including the CPI, namely cooling towers. Cooling towers are the most basic type of evaporative cooling equipment used primarily for process water cooling purposes. The following are organizations and associations that the reader can contact for further information on cooling towers and related subjects. The Cooling Tower Institute, CTI, is a non-profit organization based in Houston, Texas comprised of cooling tower users, manufacturers, and related service providers. It is probably best known for its test specifications and extensive library of information on cooling tower related subjects. The American Society of Heating, Refrigeration and Air Conditioning Engineers, ASHRAE, is an international organization which is also non profit and headquartered in Atlanta, Georgia. ASHRAE promotes standards based on extensive research, and they publish comprehensive books on the subject. Most of the weather data used by system designers comes from ASHRAE publications. They have numerous local chapters which meet regularly for educational and social events. The Refrigerating Engineers and Technicians Association, RETA, is a non profit organization based in Chicago. They publish educational materials that focus on industrial refrigeration and have numerous local chapters that meet regularly. Users and manufacturers of Evaporative Condensers should be interested in joining. The Association of Energy Engineers is similar to the above with local chapters that deal with energy issues. Cooling Towers are a common fixture in most power plants and therefore receive considerable attention from this group.

THERMAL CHARACTERISTICS

Before discussing actual equipment, a review of the thermal characteristics of evaporative cooling is presented. The latent heat of vaporization has long been used to transfer heat to the atmosphere. Our own bodies, in fact are an example of evaporative cooling, whereby our sweat glands spread water over our skin, and hence, our bodies benefit from the cooling effect that occurs when the liquid evaporates into vapor. Consider the assignment of taking a bucket of water and changing the water to vapor as quickly as possible. Several strategies can be

implemented. One approach might be to use a second empty bucket and pour the water back and forth to expose more of the water's surface area to air. The falling water could be made to splash into droplets to increase the surface area exposed to the air even further. Even more effective would be to take the bucket and dump out the water onto a large surface for maximum exposure of the water to air. To improve the operation further, a fan could be added to pass air over the water to encourage moisture laden air to exit and be replaced with new air that is better able to accept more vapor. Finally, if the quality of air could be manipulated, dry air- air that contains very little moisture- would be used because of its ability to accept greater amounts of water vapor. If the process described were modified such that new water was continually added to replace that lost to evaporation, and the water was continuously recirculated over the surface, and the runoff was continuously heated... Then we would have all the basic steps of an operational cooling system. For each pound of water that a cooling tower evaporates, it removes somewhere around 1000 Btu from the water that remains. The more evaporation that takes place, the more heat that is removed. Additional heat is taken away by the air by virtue of its temperature increase but this sensible heat exchange is minor compared to the latent component provided by the water's phase change.

In a cooling tower system, design criteria are based upon knowledge of the water flow rate, water inlet temperature, water outlet temperature, and the ambient wet bulb temperatures. Wet bulb temperature is a site condition measured by constructing what amounts to a tiny hand-held cooling tower. This small cooling tower has no heat input and is used to determine the lowest leaving water temperature a cooling tower could possibly attain accurately predicting the performance of a larger, operational counterpart. This tiny cooling tower or instrument is called a **sling psychrometer**. It places a thin film of water on the bulb of a thermometer. The thermometer is twirled in the air. After a few seconds, the thermometer begins to show a reduced temperature reading. Twirling it more will yield successively lower temperature readings until a final low temperature reading can be made after about one minute. Additional twirling serves no additional benefit. This low reading is called the **wet bulb temperature**. It is necessary to insure that the thin water film be maintained. A cotton sock connected to a small water reservoir is typically employed. Some psychrometers use a small battery operated fan so that the operator doesn't have to twirl the device in the air. Both types also have a non wetted thermometer that reads what is called the **dry bulb temperature**.

A comparison of wet and dry bulb readings allows the relative humidity to be determined from a psychrometric chart. The wet bulb temperature is always lower than the dry bulb value except when the air is already saturated with water - 100% relative humidity. This is when the wet and dry bulb temperatures are the same. The air will no longer accept water and the lack of evaporation does not allow the wetted bulb to reject heat into the air by evaporation. This situation would be

similar to operating a cooling tower at 100% relative humidity. The only rejected heat is that which is responsible for increasing the air temperature.

A single wet bulb reading will allow a prediction of cooling tower performance at that unique condition but the wet bulb changes throughout the day and year. The design wet bulb is typically determined by reviewing a chart that has been prepared by taking numerous readings in a particular area over several years and determining the maximum wet bulb readings. The wet bulb can be thought of as the heat sink temperature to a cooling tower. The lower the wet bulb, the drier the air, the more moisture it will accept and the more heat a given cooling tower is capable of rejecting.

When sizing a cooling tower, then, the highest anticipated wet bulb should be used. During the rest of the time, the cooling tower is oversized for the duty. The exiting water temperature will simply be less than design which is typically desirable. A wet bulb chart is arranged to show the frequency of occurrence. At the Charles DeGaul Airport, for example: 65 degree wet bulb is exceeded 2% of summertime hours, 68 degree wet bulb is exceeded .5% of summertime hours, and 70 degree wet bulb is exceeded .2% of summertime hours. Generally, the designer would select 70 degrees as the design wet bulb for a situation like this but some installations aren't critical allowing the use of a reduced design values and smaller cooling towers. Other installations may work only in the winter or at night when the wet bulb temperature is low. The designer must select the design wet bulb for the project. When in doubt, select the highest anticipated wet bulb temperature to insure satisfactory year around operation. The designer should only consider towers with independently certified capacities. The Cooling Tower Institute (CTI) lists towers that subscribe to their test standard STD-201. Alternately, the designer should specify a field test by an accredited independent test agency in accordance with CTI Acceptance Test Code ATC-105 or ASME PCT-23. Manufacturer's catalogs have cooling tower selection charts with easy to follow instructions that begin with the calculation of two values:

Range = Inlet temperature - Outlet temperature
Approach = Outlet temperature - Wet Bulb Temperature

These values coupled with the flow rate and wet bulb temperature allow the selection of a cooling tower. Those new to cooling towers should make several selections at different wet bulb temperatures to test how wet bulb relates to cooling tower size. It becomes clear that the tower size increases as the wet bulb rises and that the size increase becomes dramatic as the approach is in the 'less than ten degrees' area. This exercise demonstrates how to oversize a cooling tower... just use an inflated design wet bulb temperature. This is better than artificially inflating the flow rate and possibly over sizing the spray nozzles. Increasingly, manufacturers offer software to make selections easier.

Calculating the heat transfer and water evaporation rates are illustrated by the following example. A cooling tower cools 900 gpm of water from 95 to 85 °F. The problem is to determine what the heat rejection is, and also what is the evaporation rate. The heat rejection is calculated as follows:

Heat Rejection = 900 gal/min $\times$ 10 °F $\times$ 8.33 lb/gal x 60 min/hr $\times$ 1Btu/lb- °F

$$= 900 \text{ x } 10 \text{ x } 500 = \underline{4,500,000 \text{ Btu/hr}}, \text{ or}$$

Heat Rejection (Btu/hr) = Flow (gpm) $\times$ Range (°F) $\times$ 500

Evaporation Rate = Heat Rejection $\div$ 1000 Btu/lb

$$= \text{Flow (gpm)} \times \text{Range (°F)} \times 500 \div 1000\text{Btu/lb}$$

$$\times \text{ gal/8.33 lb} \times \text{hr/60 min, or}$$

Evaporation Rate (Btu/hr) = Flow (gpm) $\times$ Range (°F) $\div 1000$

A common misconception is that the cooling tower dictates the rate of heat transfer. A cooling tower simply gives up the heat it is supplied with. If the cooling tower is large, it may accomplish the job by cooling water from 90 to 80 °F. If it is small, it might cool the water in the same process from 100 to 90 °F. In either case, the heat transfer and evaporation rates are the same.

The size of the cooling tower, the flow rate and the wet bulb temperature determine the inlet and outlet water temperatures- but not the difference between them. Increased cooling tower performance can be achieved by adding surface area or by boosting the cfm.

The former is considerably more expensive than the latter inasmuch as a cfm increase can be as simple as employing a more powerful fan motor allowing increased fan speed. Cooling towers must be evaluated on a life cycle cost basis. Spending a little more for a tower that uses less horsepower or lasts longer is almost always the wisest decision.

The most common use for cooling towers is in air conditioning as the heat rejecter in a mechanical refrigeration system. The expression 'ton' is derived from this application. Its origin goes back to the earlier days when theaters, concert halls and the like were cooled with ice. Typically, this ice was harvested from lakes and stored for summer use. At its eventual destination, it would be placed in bunkers where circulated air would melt the ice and cool the air. It is hardly imaginable that such practice was widespread in many parts of the world during the early twentieth century; nonetheless, modern HVAC (Heating-Ventilation-Air-Conditioning) technology stems from this practice, and the use of early terminology has persisted through modern times.

With the introduction of mechanical refrigeration, the term 'ton' was retained. The owner could now buy a system capable of providing the equivalent capacity of however many tons was needed. Since one pound of ice absorbs 144 Btu when melting, one ton of ice melting over a period of 24 hours has a heat transfer rate equivalent to the following:

$$\text{Heat Transfer Rate} = 2,000 \text{ lb} \times 144 \text{ Btu/lb} \times 1/24\text{hrs} = \underline{12,000 \text{ Btu/hr}}$$

Thus, when the occupants of the room experience heat removal at the rate of 12,000 Btu/hr, they are enjoying one ton of cooling. The mechanical refrigeration system utilizes a compressor that adds its heat energy- basically the motor horsepower- to the refrigerant. The cooling tower must reject not only the 12,000 Btu/hr/ton from the space but also the heat of compression as well. This added load typically amounts to about 3,000 Btu/hr/ton for air conditioning systems. So, while the occupants are enjoying 12,000 Btu/hr/ton heat removal, the cooling tower is rejecting heat at a rate close to 15,000 Btu/hr/ton. This gives rise to the term 'Cooling Tower Ton' which is defined as 15,000 Btu/hr. This definition is only valid for typical air conditioning conditions. Another very common term is 'nominal capacity'. It also has its roots in air conditioning and involves the following assumptions: The cooling tower will circulate water at the rate of 3 gpm/ton. The water enters the tower at 95 °F and exits at 85 °F. The design wet bulb temperature is 78 °F. Evaluating cooling tower capacity at the nominal conditions is an easy way to determine which tower is larger (the term'bigger' is often used) when comparisons are made. Someone employing mechanical refrigeration at 95/85 °F condenser water conditions in a city that actually has a 78 °F wet bulb temperature can use the nominal ratings as an accurate prediction for the cooling tower performance. A reduced wet bulb temperature allows the use of a smaller cooling tower. The converse is true - at wet bulb temperatures above the nominal 78 °F value... the nominal rating of the cooling tower on a 300 ton project at 80 degree wet bulb will be more than 300 tons (actually, about 360 tons).

Absorption refrigeration is another method of making chilled water for air conditioning. Instead of a compressor, the design utilizes heat energy to increase the pressure of a refrigerant. An absorption system by its nature requires a cooling tower that will remove about 50% more heat than a mechanical refrigeration system of the same capacity. The cooling tower flow rate, range, or a combination of the two must be increased so that their product is about 50% more than that for a mechanical refrigeration system.

Altitude has an effect on cooling tower performance but in a unique way. Air handlers, air cooled condensers and the like are typically made to operate at higher speeds (or, with a steeper fan pitch) as altitude increases in order to maintain the

same mass flow. As an example, air at 5,000 ft. is approximately 17% less dense than at sea level and the fan speed increases by the same amount. A cooling tower designed for operation at sea level will work just fine at 5,000 ft elevation without modification. This is because air at reduced atmospheric pressure will accept increased amounts or water. The increased ability for the air to accept more water offsets the reduced air mass resulting in a small net gain in capacity at altitude. This is why manufacturers do not make altitude corrections with their small, package towers.

DESIGN CONFIGURATIONS

Cooling towers are classified according to the method by which air is introduced to the tower. The principal types are atmospheric spray, natural-draft, mechanical-draft, deck-filled, spray-filled, coil shed and hyperbolic towers. Most industrial cooling tower installations are field-erected units designed for specific thermal characteristics. In an atmospheric spray tower the air movement is dependent on atmospheric conditions and the aspirating effect of the spray nozzles. Natural-draft cooling tower operation depends on a chimney or stack to induce air movement. Mechanical-draft cooling towers utilize fans to move ambient air through the tower. Deck-filled towers contain tiers of splash bars or decks to assist in the breakup of water drops to increase the total water surface and subsequently the evaporation rate. Spray-filled towers depend only on spray nozzles for water breakup. Coil shed towers are comprised of a combination structure of a cooling tower installed on top of a substructure that contains atmospheric section coils. Hyperbolic natural-draft cooling towers are typically large-capacity systems. There is also a separate class of towers based on a dry cooling principle.

Cooling towers are broadly classified on the basis of the type of draft: natural draft (natural convection), mechanical draft (forced convection) and mechanical and natural. Further distinction is made based on (1) the type of flow; i.e. - crossflow, counterflow, cocurrent flow; (2) the type of heat dissipation-wet (evaporative cooling), dry, wet-dry; and (3) the type of application-industrial or power plant. Each of the major types of cooling towers has a distinct configuration. The major designs are summarized in Figures 1 through 8 and a brief description of each follows.

In an atmospheric spray towers, the air movement depends on atmospheric conditions and the aspirating effects of the spray nozzles (refer to Figure 1). In comparisons, a natural-draft cooling towers, as illustrated in Figure 2, depend on a chimney or stack to induce air movement through the tower. Both design configurations are very common in the power industry throughout the world.
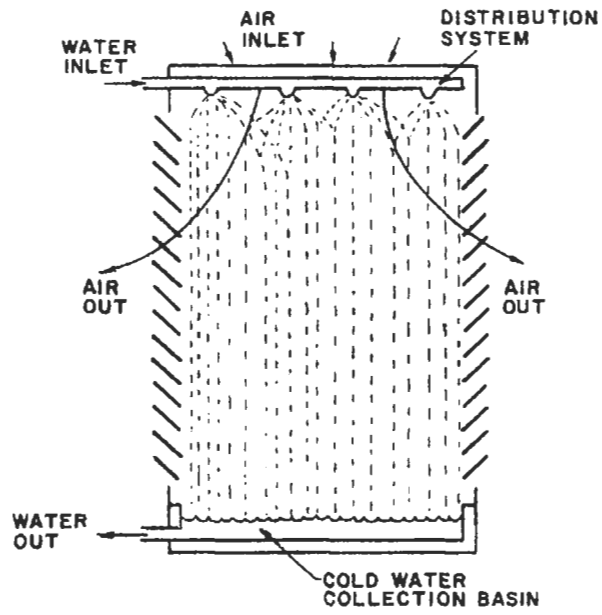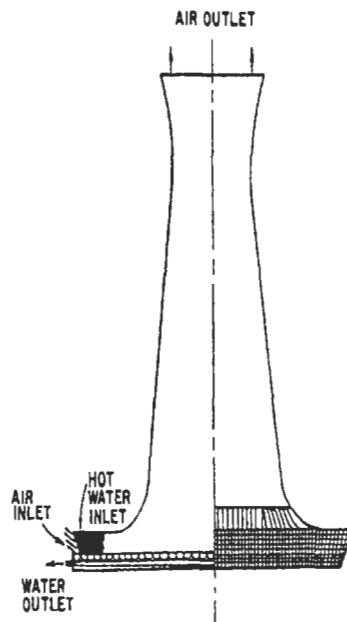
Figure 1. *Atmospheric spray tower.*

Figure 2. *Hyperbolic cooling tower.*

Mechanical-draft cooling towers use a fan to move ambient air through the tower. They can be subdivided into two different types-indirect or forced air towers, depending on whether the air is pulled or forced through the tower. At the same time they can be grouped in crossflow or counterflow, depending on the relative movement of air and water. In the crossflow type, air generally travels horizontally across the failing water, while in counterflow, it travels vertically upward through the falling water. Figures 3 and 4 provide some examples.

Another type of crossflow cooling tower is the wet-dry tower, which consists of a normal crossflow tower over which a few air coils are placed. The hot water is first cooled by an air cooled heat exchanger and then drops to the wet cooling tower where more cooling is obtained by the evaporative mechanism. Figures 5 and 6 provide examples. In contrast, deck-filled towers contain tiers of splash bars or decks to aid in the breakup of water drops to increase the total water surface and, subsequently, the evaporation rate.
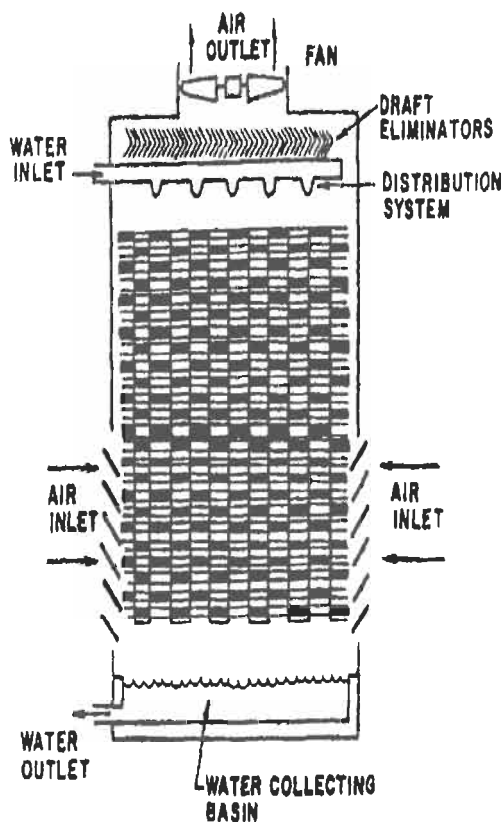
Figure 3. *Counterflow cooling tower.*
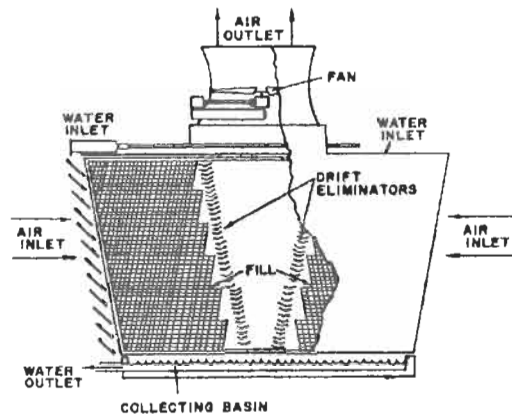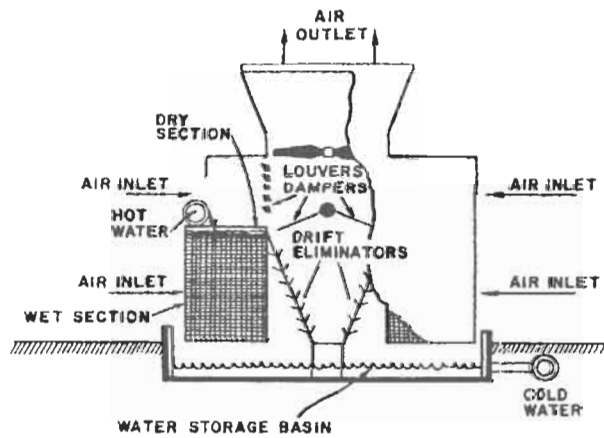
Figure 4. *Crossflow cooling tower.*

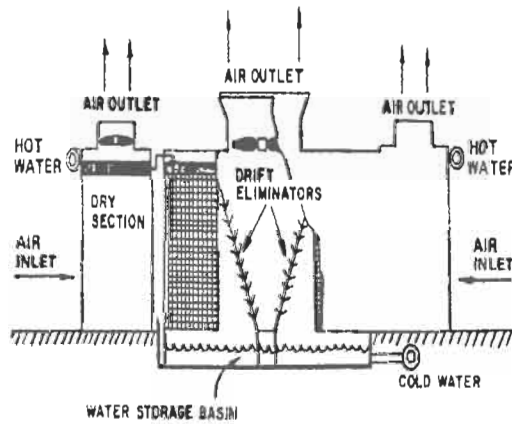**Figure 5.** *Single structure type wet-dry cooling tower.*

Figure 6. *Wet-dry cooling tower.*

Other common design configurations include:

- Spray-Filled Towers - These depend only on spray nozzles for water breakup.

- Coil Shed Towers - These are composed of a combination structure of a cooling tower installed on top of a substructure that contains atmospheric section coils (refer to Figure 7).

- Hyperbolic Towers - Natural-draft cooling towers with a hyperbolic configuration are usually constructed of concrete, have a large dimension and, consequently, large capacities. They are generally used in power plants. Figure 2 also illustrates this design.

Natural-draft cooling towers evolved from spray ponds. The simplest and earliest design consisted of a small water spray pond surrounded with walls having inwardly sloping louvers. Figure 8 shows a cross section of a simple cooling tower formed by enclosing a spray pond with louvered walls. In this type of design, the air changes direction in passing through the louvers as it leaves, and the suspended water droplets impinge on the louver slats, deposit outward and drain back into the tower basin. This design is improved by utilizing low-pressure water sprays at the top, positioning horizontal decks in the tower, and arranging the walls so that the air enters horizontally and is discharged vertically. The vertical upward movement of air tends to slow the downward velocity of the water droplets, thus increasing the effective surface area for any given water loading. The fill material is usually staggered so that water droplets fall through a distance of only a few feet before striking another surface. Typical grids are made of redwood strips railed to 1 x 2 inch stringers.
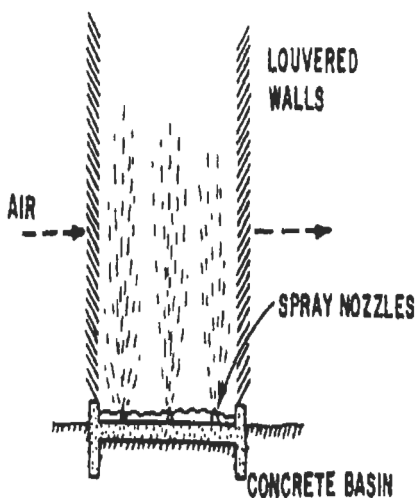

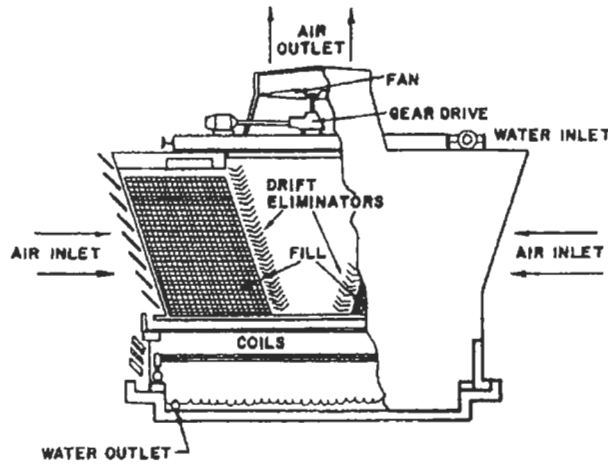**Figure 7.** *Simple configuration.*

Figure 8. *Coil shed cooling tower.*

The increasing temperature of the upwardly moving air stream induces circulation by thermal convection. This is a favorable condition if the wind velocity is very low. Towers are generally placed in aside-by-side arrangement (i.e., in a row) at right angles to the direction of prevailing winds. Hyperbolic towers are an important class of cooling towers. Early designs consisted of a cylindrical configuration. Later designs used a pair of truncated cones. Today's design consists of a hyperbolic shape. The advantages of this configuration include (1) superior strength, (2) they are a close match to the natural flow of air through the tower shell, and (3) the need for fewer materials of construction than for earlier shapes (less total volume is required because the hyperbolic shape of the shell provides good strength, so a thinner shell thickness can be used in comparison to older designs). The performance of a natural-draft tower is characterized in terms of a duty coefficient (C_D), which defines the overall capabilities of a tower under all operating conditions. The draft is due to the difference between the density of the air leaving the tower and that entering the tower, and to the aerodynamic lift of the wind passing over the top of the tower. Increases in loading, cooling range and humidity all lend to improve cooling tower performance. As already noted, the two basic types of hyperbolic towers are counterflow and crossflow, as shown in Figure 9. Of the two operations, counterflow (or countercurrent) provides the more efficient heat transfer mechanism because the coolest water contacts the coolest air initially. In the crossflow operation, air flow is normal to the water movement and fill is needed to transfer a given quantity of heat. These designs have fill in a ring outside the tower. This produces a lower water pumping head than in the counterflow. Fill inside a counterflow must be spread over a much larger area. Thus, crossflow units have shallower depths and vertical water risers have shorter pressure drops through the fill (much less than in a counterflow tower).
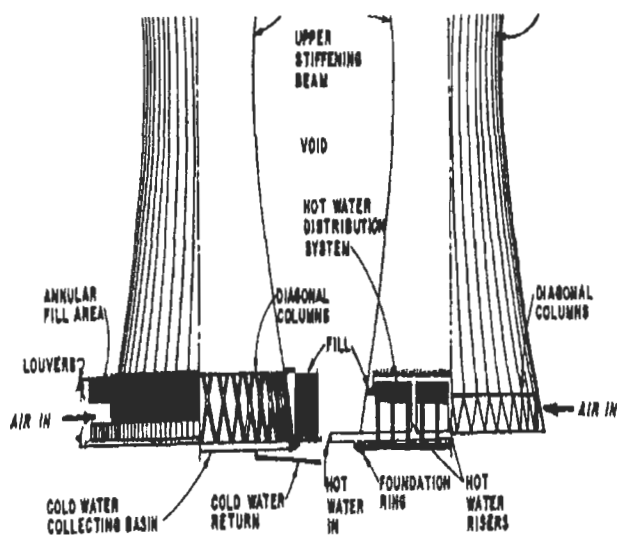
Figure 9. *Compares cross flow and counterflow towers.*

COMPONENTS AND MATERIALS OF CONSTRUCTION

Redwood was an early construction material for cooling towers because of its natural tendency to inhibit decay. As supplies of good quality redwood diminished, douglas fir was pressed into service. Its superior strength allowed for the use of fewer structural members but it deteriorated more easily. Treatments were developed to kill the decay causing micro organisms that depleted the wood. The lumber was essentially dipped preserving chemicals which were toxic in nature. The process was enhanced by incising and pressure treatment. Incising is a procedure where small longitudinal incisions are made into the wood to encourage the acceptance of chemical treatment. Pressure treating has the wood placed in a vessel that is first evacuated to remove moisture from the wood followed by the introduction of chemicals under pressure. Simultaneous with the greater reliance on fir was the increased use of factory assembled steel cooling towers in ever increasing sizes. Galvanized steel performed well with the water treatment chemicals of the day, which were predominantly chromates and acid. Acid was used to lower the pH allowing higher cycles of concentration of the dissolved solids while chromates quelled the rampant corrosion that would otherwise occur. Asbestos cement board casing side panels were popular on field erected cooling towers. Asbestos was also found in the wet deck and sealing compounds of many towers.

Increasing environmental concerns in the 1970's brought an end to the use of chromates except for a few very large facilities where they were removed from the discharge water at site treatment facilities. Without chromates, the low pH water was very corrosive and many cooling towers and piping systems were ruined in short time. Substitutes have never lived up to chromates for effectiveness and cost. Wood towers didn't escape intense environmental scrutiny. The potential hazards of wood treatment chemicals became more apparent causing revised formulations and tighter controls both leading to increased costs. Asbestos also came into disfavor and was quickly phased out of cooling towers as the manufacturers became more aware of the potential health and financial liabilities. Type 304 stainless steel (SST) became more popular as the corrosion potential increased. Manufacturers simply substituted stainless steel for galvanized steel components. Due to cost constraints, just the cold water basin was typically upgraded to SST. There were some unfortunate occasions where galvanized and stainless steels were fastened together below the water line causing rapid deterioration of the galvanized steel at the joint from galvanic corrosion. Anyone considering mixing these materials must pay attention to the surrounding materials, particularly the fasteners. Such joints should never occur below the overflow level of the cooling tower.

Specifiers will sometimes call for type 316 SST. This is generally acceptable for nuts, bolts, and some small sub assemblies but it is largely incompatible with the tooling used by the manufacturers. It is also difficult to form. For these reasons, it is largely unavailable. The galvanized steel cooling tower has remained the factory assembled standard to this day. The thickness of the steel has steadily declined with more economical designs but the thickness of the zinc layer has steadily increased to a current standard of G235. (or, 2.35oz. of zinc per sq. ft.) from a 1970's standard of G90 (.90 oz/sq.ft.). This thickening of the sacrificial zinc layer has a very beneficial effect on cooling tower life. Various enhancements to the galvanized steel in the form of barriers have been employed by some manufacturers. Their suitability largely depends on the local water quality.

Concrete can be an excellent construction material for basins- even side walls, fan decks, discharge stacks, and mechanical support beams. Its use beyond basins, however is not typically justified for commercial applications. Extensive concrete construction is used for architectural reasons- where the tower is disguised to look like or blend in with a building- or, the cooling tower is designed as a structure with a life expectancy equal to the facility it serves such as a hospital or university. Pultruded fiberglass is increasingly replacing steel in structural applications. These are composites with precisely located glass fibers that make the parts very strong. The process allows the addition of surface treatments that limit ultra violet degradation- an important requirement for cooling tower duty.

The wet deck or surface, is the heart of most cooling towers. Generally, it takes the form of PVC (polyvinyl chloride) plastic film type surface. Water is made to spread out on this surface maximizing it's contact area with air to encourage evaporation. It consists of individual vacuum formed sheets with proprietary patterns of ridges,

bumps and wrinkles. When arranged vertically (side-by-side), the individual sheets space themselves apart leaving passageways for water and air. The sheets can simply press against each other or be glued together. Edges can be folded for increased strength. A block of glued together film type fill can be placed on a table top and observed. If a marble were dropped through the fill it would follow one of the channels that are formed between the adjacent fill sheets. These channels- or flutes- are typically at an angle to vertical to increase the residence time of the water as it falls through the wet deck. The goal of the film type wet deck designer is to maximize air/water contact while minimizing air flow pressure drop. Reduced flute sizes increase thermal capacity limited by air flow pressure drop and clogging. Typical clean water applications such as air conditioning can have marble sized flutes. Dirty water applications like steel mills, on the other hand, may require golf ball or base ball size flutes.

Crossflow and counterflow wet decks are designed differently. Tile fill is a bullet proof approach to wet deck. It is suitable for clean to moderately dirty water and has extraordinary longevity. Heat transfer efficiency is less than that for film type wet deck, however, requiring more volume or more fan horsepower for equivalent capacity. Splash bars are another method tailored to extremely dirty water applications. Instead of spreading the water into a thin film, the approach is to have the water splash into droplets as it cascades through the tower splashing off successive splash bars. Clearly, the total surface area of all the water droplets is far less and the thermal capacity is diminished versus film type fill. On the positive side though, considerable debris can be tolerated and cleaning is relatively easy. The ultimate in dirty water towers is the 'spray fill' design. Here, there is no fill at all. Water simply sprays into the empty plenum area of a tower. This design is limited to counterflow type towers.

Eliminators are used to remove water droplets from cooling tower discharge air by imparting several rapid directional changes. The heavier water particles collide against the eliminator and drain back into the tower. Superior eliminator designs limit escaping water droplets of the recirculated flow rate while imparting minimal pressure drop to the airstream.

The Spray tree is used to distribute water over the wet deck in counterflow cooling towers. It can consist of a single header fitted with spray nozzles or, it can utilize spray branches with nozzles for wider coverage. Spray nozzle designers seek minimal pressure requirements and uniform coverage over wide flow ranges.

Hot water basins are used to distribute water in crossflow towers. Here, water is pumped to an open pan over the wet deck fill. The bottom of the pan has holes through which water is distributed. Manufacturers will fit specially shaped plastic drip orifices into the holes to give the water an umbrella shape for more uniform distribution. Different size orifices are used for different flow rates. Ideally, the basin will be almost full at maximum flow. This way, sufficient depth is retained for good water distribution as turn down occurs. The turn down ratio can be extended by the addition of hot water basin weirs- a pattern of baffles perhaps 2

inches tall fastened to the basin floor- that insure good water distribution by selected nozzles at reduced flow. As full flow is restored, the water overflows the weirs to again engage all available orifices. Cold Water Basins collect cooled water at the bottom of the tower. They are an integral part of factory assembled designs and are built in place- typically of concrete- for field erected towers.

A make-up valve replaces water that exits via evaporation and bleed with fresh water. It operates somewhat like the valve found in a conventional toilet tank but is larger and more heavy duty. Like toilet tank floats, they can function mechanically or hydraulically. Cold Water Basin Heaters address freeze-up for cooling towers in cold climates. They are electric immersion heaters installed below the water level that add sufficient heat to prevent freeze damage. A 1,000 ton cooling tower may use something like 30 KW. This is equivalent to 30 KW x 3,415 Btu/hr/KW x 1/ (12,000 Btu/ hr/ ton) = 8.5 tons. Some designers expect immersion heaters to heat the system water as an aid to cold system start up but sump heaters are clearly inadequate for this task. Water cascading through a 1,000 ton tower- even with the fan off- will easily reject more heat than a small sump heater can possibly add. Sump heaters should be interlocked with the system pump and only operate when the pump is idle. Properly configured controls will include a thermostat typically set to energize the heater when the sump water temperature is below 40 °F. Steam or Hot Water Coils can also be employed in lieu of electric heaters.

It is never appropriate to add any type of anti-freeze solution to an open cooling tower. Closed (fluid cooler) systems, however, can be protected from freeze-up by the addition of ethylene glycol or other fluids. Fluid cooler casing sections can also be insulated to reduce heat loss thereby protecting the coil from freeze-up. Counterflow, blowthrough towers tend to be more popular as the freeze potential increases. Crossflow towers tend to freeze water on their air inlet louvers under extreme conditions. Fans (propeller type) can be arranged to reverse direction on such towers to melt ice. This process should never be automated. Instead, the operator should weigh the situation and reverse the fan only as long as required. The designer must select components suitable for reverse rotation. Fan discharge dampers are a capacity control accessory item for centrifugal fan cooling towers. They fit in the fan scroll. In the open position, they are much like a thin piece of sheet metal in a moving airstream oriented parallel to airflow. The airstream doesn't know its there. As the dampers close- the sheet metal becomes less parallel to airflow- turbulence disrupts the air stream. Airfoil dampers essentially ruin fan housing efficiency to achieve a reduction in airflow. Dampers can set and locked when a manual locking quadrant is specified but it is more common to use electric or pneumatic actuators that close the dampers as the exiting water temperature becomes too low. While reducing airflow is the correct method of reducing capacity, dampers are not the best approach. They offer the poorest energy savings and the actuating mechanisms tend to fail long before the average cooling tower life span.

System designers often think dampers block airflow and are suitable to prevent back drafts in idle towers. This is not the case. Airfoil dampers simply hamper fan housing efficiency- they do not block airflow. Air Inlet Screens are always part of blow through, counterflow towers to protect people from rotating equipment . Some designs can be a hazard when accessible from the underside and require the specifier to call out additional screening. They can be a worthwhile accessory when there are nearby trees even when not required for safety reasons. Air inlet screens should be eliminated on towers utilizing inlet ductwork. Inlet ductwork may also make it necessary to block extraneous air entry such as from the underside when towers are elevated.

A vibration cutout is a control device used to shut down the fan motor when excess vibration is sensed. They can be used on any tower with a fan motor either by choice or by code although they are only practical for towers employing large propeller fans. Typically, centrifugal fans do not fail in a catastrophic mode; Similarly, small prop fans don't cause enough damage to require such devices. Ladders and handrails are also necessary components for large field erected cooling towers and make sense on some factory assembled designs. Often, just a ladder makes more sense. Or, nothing at all on small towers. Internal Ladders, Walkways, Platforms, etc. should be evaluated on a job by job basis.

USE OF FANS, MOTORS, AND DRIVES

Cooling tower fan motors must be properly selected for long life and trouble free operation. In addition, it is important to match the motor's enclosure to the application. Motors in draw through applications that are mounted directly in the air stream, for example, experience tough duty and must be of the totally enclosed type. Motors are categorized as follows:

- TEAO - Totally Enclosed Air Over where the motor has large cooling fins and depends on the cooling tower air stream for air movement. The motor shaft protrudes from the enclosure at one place only.

- TEFC - Totally Enclosed Fan Cooled where the shaft extends from both ends with a cooling fan attached to one with a shroud that directs air over strategically located cooling fins. This motor is more commonly used when the motor is outside the air stream as when driving a gear box with a drive shaft or on blow-through towers.

- ODP - Open Drip Proof where there are openings to the windings through the enclosure and a cooling fan inside that causes air to flow through the motor. ODP motors should never be installed in a discharge air stream. They can be placed in the inlet air when located in such a way as to be protected from

splash out and rain. TEFC motors should be the minimum standard. TEFC motors in the discharge air stream with the shaft pointing down- as with most belted applications- have an increased possibility of moisture entering the motor around the cooling fan.

In addition, the small motor fan attempts to blow air down over the motor and is no match for the much bigger cooling tower fan drawing air up and around the motor. The small fan in this case is useless- even counterproductive. This application needs a TEAO motor. Cooling tower manufacturers sometimes purchase special motors tailored for their application... special grease, seals, slingers, weep hole locations, epoxy coatings, etc.. Such features make an OEM replacement more desirable than an 'off the shelf' replacement.

One advantage of having the motor in the air stream is the generous cooling that it receives. A motor rated at, say, 60hp in a 'normal' application may be capable of providing a continuous 72hp without any difficulty because of this cooling. This is why it is important to size conductors, fuses, starters, etc. based on actual motor amps (which the manufacturer should provide in his quotation) and not from standard application charts.

Large towers often have the motor mounted horizontally connected to a right angle gear drive. The motor can be closely coupled- in the air stream- or connected with a drive shaft with the motor outside the air stream. Maintenance personnel typically prefer the external TEFC motor- when available- for its easier access. Cooling tower fans- like all fans- operate in accordance with the fan laws one of which states that the horsepower required to drive a fan increases to the cube of fan speed.

$$HP_2 = HP_1 \ (RPM_2 \ / \ RPM_1)^3$$

As an example, consider the speed of a fan that is increased by 10%. Let's determined the revised horsepower requirement.

$$HP_2 = HP_1 \ (RPM_2 \ / \ RPM_1)^3 = HP_1 \ (1.1 \ / \ 1.0)^3 = 1.33HP_1$$

The horsepower increased 33% while the speed went up by only 10%. Similarly, slowing the fan by a small amount causes a marked reduction in the horsepower and amperage draw. It is apparent that speeding up a cooling tower to increase its airflow and capacity must be carefully approached. Aside from possibly exceeding the maximum safe speed for the fans or causing the increased airflow to 'fling' water past the eliminators - a phenomenon referred to as 'spitting'- the required horsepower climbs very quickly requiring oversized motors with attendant energy costs for only modest capacity gains. This fan law demonstrates another important fact, namely, that fan motors have small torque requirements at start up and don't begin to really work until they get near top speed. Fan motors, therefore, do not need special starting schemes such as part winding or Y-start, Delta-Run. Fan

motors are simply started across the line. Occasionally, motors show optional starting features on their name plates causing operators to seek out the special starters depicted for no reason. This is simply a case where the motor manufacturer stocks motors with a wide application range. Cooling tower fan motors are often used to modulate air flow through a cooling tower to kill off excess capacity during periods of low load, or especially during winter operation. This capacity reduction can be achieved with fan cycling, multi-speed motors, extra motors, or variable speed drives. Fan cycling (turning off fan motors) works well when a tower has numerous fan motors. If there are four fan motors, for example, turning one motor off reduces the capacity by about 1/4. This is an easy capacity control method but doesn't work well when close temperature control is required resulting in frequent motor starts. As a rule of thumb, six starts per hour should be considered maximum. Excessive starting causes heat build up and insulation failure. Prolonged "pump on, fan off" operation is not good and should be avoided. Two speed fan motors are available as either single or dual winding. The single winding motor has its entire winding active at low or high speed. The winding is simply reconfigured by the starter as either 8-pole or 4-pole (900rpm or 1800rpm). Low speed is always half of full speed. These motors are wound for a specific voltage- most often 460v. The two winding motor has two separate and distinct windings- one for low speed and the other for high speed. It is possible for one winding to fail leaving the other intact but such occurrences are rare. Typically, a faulty winding takes out its neighbor. And, since the motor has to be removed for repair anyway, there is no real standby advantage to such a motor. One advantage of a two winding motor is that the speed ratio is not necessarily 2:1. Common speeds are 1800/900 and 1800/1200. These motors are also wound for a specific voltage. In general, single winding motors cost less but their starters cost more. Conversely, two winding motors cost more and their starters are less expensive. In the end, there is little cost difference. Single winding motors are more likely to be stocked and are far more popular. Either should be specified as variable torque.

Pony motors are additional, small motors connected to the same fan shaft. They are typically about 1/4 the size of the full size motor. Pony motors do not lend themselves to gear box applications and are, therefore, primarily applied to belt drive applications. The big motor operates when full capacity is required and the small motor simply free wheels. At reduced capacity, the small motor operates and the big motor spins freely.

The appropriate drive ratio is selected for each motor so that it is fully loaded when in operation- a distinct advantage over two speed, variable torque motors where the available horsepower is proportional to the square of fan speed while the required fan horsepower varies as to the cube of fan speed (from the fan law). As an example, a fan motor that can produce 40 hp at high speed can produce 10 hp at low speed while a fan that requires 40 hp at high speed only requires 5 hp at half speed. As a result, 1800/900 rpm motors are always 100 % oversized at low speed. And, since the motor usually operates at low speed most of the time, the

inefficiency of the lightly loaded motor is noticeable. The pony motor also has the advantage of allowing one motor to be removed for servicing while the other remains on line. Plus, such simple, single speed motors are readily available in the event of a breakdown. Electrically, the pony motor arrangement is equivalent to a two winding motor. The two windings physically reside in separate motors.

Questions arise as to the 'idle' motor acting as a generator when rotated by the active motor. Aside from other differences between motors and generators, the fact that there is no excitation current means that there can be no output. The only losses seen by induction motors are from windage and belt flexure and are so small as to be virtually undetectable.

Single phase motors do exhibit operational problems in pony motor applications. The capacitors in these motors store energy and the motors resist external attempts to rotate them. Single phase motors can be made to work on pony motor applications by splicing into the capacitor circuit and connecting it in series with an open auxiliary switch in the active motor starter.

Examination of the drive ratios shows that if both motors have 1800rpm synchronous speeds that the pony motor is made to operate near 3600rpm when the big motor is operating. This is typically not a problem as to rotor balance or bearing duty because manufacturers make 3600 rpm versions of these same motors. Nevertheless, the duty should be checked and if this is a problem, the small motor can be changed to a 900 rpm model and the synchronous speeds of each motor will not be exceeded.

Two speed motors and pony motor arrangements both require a time delay that prevents low speed or pony motor operation until approximately 15 seconds after high speed operation. This insures that the low speed winding is not energized while the motor is rotating faster than its synchronous speed. For example, an 1800 rpm pony motor rotates at about 3600 rpm when the 'big' motor is operating. If the 'big' motor is switched off and the pony motor switched on immediately, it would attempt to operate at its 1800 rpm synchronous speed but would already be rotating closer to 3600rpm. The conflict will cause a deceleration of the rotating components so severe as to possibly cause damage. The 15 second time delay insures the motor is rotating under its synchronous speed when energized. It will then gently accelerate to full speed. Fifteen seconds is a starting point; The time delay can be reset to a lower value appropriate for each project.

Variable speed drives are the ultimate in capacity control but introduce a level of sophistication that may not be required. Projected fan motor energy savings make their use attractive, however there are other considerations. Often the excess energy consumed by a single speed motor is more than offset by increased system efficiency from the reduced water temperature provided by the cooling tower. When used, VFD's and two speed motors should generally be set to provide the coldest temperature that the system will tolerate before reducing motor speed. VFD's can, however, be very helpful in noise sensitive applications. Soft starting and gradual speed changes make cooling tower noise less noticeable to critical

neighbors. Towers that operate at extremely light loads can also benefit from VFD's. The VFD will keep a motor running with a positive air flow through the tower. This avoids excessive 'motor off' operation and attendant water 'splash out' problems. Note also that extremely low speeds can defeat the 'sling' lubrication employed in gear boxes requiring the drive to be programmed to avoid low speeds. Alternately, an electric oil pump can be applied to insure adequate lubrication at all speeds.

A common problem encountered is 'windmilling'. This is when the cooling tower is baffled in such a way as to allow air to pass in a reverse direction past a supposedly idle fan when its neighbor is in operation. In this situation, the 'idle' fan rotates backwards. This causes numerous problems not the least of which is tremendous stress on the drive components when they attempt to start while rotating in a reverse direction. Economic design is often responsible for the omission of baffles. The designer should not assume that just because there may be multiple motors that they can all be operated individually. Anti windmilling devices- essentially one way clutches- are available to some applications but are not necessarily the best solution. It is better to baffle the tower properly so that each fan can operate independently. When replacing fan motors, be sure to match all the nameplate characteristics such as hp, rpm, voltage, phase, frame size, enclosure, service factor, insulation class, group, etc.. Also check that the conduit box is located in the same place. NEMA -the National Electrical Motor Association- provides standards for motor manufacture. Motor frame size is one important NEMA standard. A 284T frame motor from one manufacturer, for example, will have the same bolt pattern, shaft elevation, diameter, key size, etc...among all manufacturers. UL -Underwriters Laboratories- examines components from the various motor manufacturers and publishes a list of UL recognized motors. This prevents UL from having to check each motor that they encounter when evaluating equipment in the lab. Most cooling towers are not UL listed. This is because the smoke and debris that would result from a motor failure is not directed into occupied spaces. UL. listing is therefore not required.

Cooling tower fans can be directly coupled to the motor, or, connected via gear boxes or V-belts. Direct drives are limited to small diameter fans. Otherwise, tip speeds would be too great- even with 900rpm motors. Direct drives are, therefore, generally found on small cooling towers; or, medium size towers with numerous motors. V-belts are used on the full range of factory assembled towers- from less than 10 tons to over 1,000 tons. They allow the manufacturer to easily tailor the speed of fans through the judicious selection of sheave (pulley) diameters. Fixed pitch prop fans can be used. V-belt drives do not require precise alignment and work fine after the rigors of shipment and rigging. Belts and sheaves come from numerous manufacturers.

Gear boxes are used on just about all large field erected cooling towers and many factory assembled towers- all of the propeller type fan, draw-through type. Almost all are right angle gear drives with the input shaft horizontal and the output

vertically 'up'. The designer selects the ratio closest to his/her requirement based on the fan being used, noise levels and cost. Limited ratios are available and the fan blades must be 'pitched' to the proper angle to achieve the desired performance for the specific project- typically not a problem since large fans are built on site anyway.

Speed reducers should be right angled gear drive type specifically designed for cooling tower service. Gears should be spiral bevel or helical type designed in accordance with the Cooling Tower Institute and American Gear Manufacturer's Association Standards with a minimum service factor of 2.0 based on motor horsepower. The gear should be suitable for both forward and reverse operation. Oil fill and vent lines should be extended outside the fan stack and provided with an oil level sight glass to facilitate routine inspection and maintenance. Fan motors can connect directly to the gear box or through a drive shaft. Either way, motor and gear alignment are critical and mountings must be solid. Various manufacturers make flexible couplings specifically for difficult cooling tower duty. It is in the cooling tower manufacturer's best interest that these items work properly and be trouble free.

As with any mechanical equipment, cooling towers can generate objectionable noise and vibration. Large installations are typically laid out by experienced designers. They gather equipment away from noise sensitive areas, add concrete walls as barriers and employ acoustical consultants when necessary. Sound problems are generally infrequent but can be difficult to manage. Minimally designed condominiums or industrial plants that boarder residential areas are typical. Sound complaints don't always involve rotating machinery. The designer must strive to reduce changes in noise level in noise sensitive situations. Motors starting or changing speed, belts squealing and the like all draw attention to the tower. Variable speed drives or 'soft starters' can help to minimize these problems. Two identical towers side by side can exhibit the phenomenon where sound pressure levels are slightly in and out of phase causing an annoying addition of sound pulses- like that of a twin engine turbo prop commuter plane. Intentionally making the fans operate at different speeds through the programming of VFD's- or simply changing one of the sheave diameters slightly on a belt drive tower can help. Factory designed sound attenuators that attach to the tower can be helpful in difficult situations; However, they tend to be expensive and generally require more fan hp. So, it is better to first consider oversizing the tower and slowing the fan before employing attenuators. Vibration can also be annoying and difficult to solve. When towers are on floors or rooftops with people below, it is wise to employ spring type vibration isolation. The amount of static deflection is an indicator of isolation efficiency- generally 1" minimum and 2" maximum. Another consideration when using vibration isolators is to first mount the tower on a sturdy frame. Then, place the isolation underneath. This is especially critical for multi-cell towers. All cells must be mounted on a single frame before being isolated. Otherwise, the reduced water weight that occurs when valving off and draining one cell allows its springs

to extend causing tremendous stresses to flume boxes and interconnecting piping. Rubber pads offer a small measure of vibration isolation and work well when placed under a uniformly distributed load. However, some tower designs have highly concentrated load points that will cut through the pads. Worse yet, the underside of some towers can become distorted when they exhibit variable loading characteristics- much like a person attempting to sleep on an overly soft mattress. Rubber pads should be avoided unless the designer is sure they are appropriate. After taking the effort to isolate the tower, it is mandatory that the piping have flex connectors.

WATER TREATMENT SERVICES

Water treatment is a necessary adjunct to evaporative systems. Water quality must be maintained to prevent scaling, corrosion and biological fouling or attack. Scale and corrosion are generally thought to be diametrically opposed to one other. Reducing scale build up, for example, exacerbates corrosion and vise versa. The biological aspect of water treatment comes from living organisms that thrive in the recirculated water and wetted surfaces. Bacteria, slime and algae can foul heat exchanger surfaces and in some cases attack and destroy system components. Chemical treatments address biological issues separately from scale and corrosion. Scale formation has its root in the evaporation of water. Evaporated water exits the system as pure vapor leaving the solids behind. The replacement (make-up) water introduces more solids which continually increase the solids concentration in the recirculated water. Left unchecked, the system would reach a point where the water could not hold all of the solids in a dissolved state. They would begin to precipitate out of solution as scale. The necessary steps to combat scale differ by geographic location. Evaporating enough water to make the solids increase to twice their initial value is a two fold increase in solids content. Clearly, water that has few initial dissolved solids can attain a very high number of 'cycles' before the solids precipitate from solution. Conversely, water with high initial solids can only be 'cycled' a small amount before precipitation occurs. In either case, a saturation point will be reached where the cycles cannot be increased. Every particle that dissolves is offset by another particle that comes out of solution as scale. The water treatment service provider is aware of the dissolved solid content of the water in his/her area and knows how many cycles are acceptable. When in doubt, a make-up water sample can be taken to demonstrate the quality of the water entering the system. Each constituent is examined against a maximum allowable concentration. Some locations may require that the cycles be set based on keeping some other constituent- like silica- below a maximum threshold value. Recognizing the concept that the fewer the initial solids the better, some operators are tempted to use soft water as make-up theorizing that since most of the solids are removed, the cycles

can be allowed to reach astronomical levels without scale formation. This approach is ill advised without the input of a competent water treatment expert who is capable of combating the excessive corrosivity of such water. In addition, the use of water treaters adds chemicals that allow the water to retain increased solids.

Water treatment controls are set to maintain the design value. This could be a 'continuous bleed' where a set portion of the recalculated water is intentionally wasted to the drain. The make-up float valve introduces fresh water to replace that which is evaporated and bled. The new water mixes with system water diluting the solids concentration. While initially inexpensive, the continuous bleed is rarely used and, in fact, is often illegal. Here, a valve is set to waste water to the drain at a rate necessary to maintain the design cycles at maximum evaporation. The problem is, towers typically aren't called upon to evaporate water at the maximum rate most of the time allowing the cycles to plummet, wasting water. More common is the use of a 'conductivity monitor' which operates on the principle that the conductivity of water increases in direct proportion to its solids concentration. The device is first used to measure the conductivity of the make-up water and then set to initiate a bleed cycle when the system conductivity reaches a value equal to this initial reading x cycles. Scale and corrosion inhibitors are typically injected into the system as it bleeds. Liquid chemicals are introduced by small, adjustable, plastic, positive displacement pumps that meter precise dosages.

The tendency for any system to grow biological material depends on several factors. Cooling tower design is one. Crossflow towers and counterflow towers without louvers, for example, tend to grow more algae due to the increased amounts of sunlight in contact with the system water. Water quality also comes into play. Make-up water that is reclaimed from a sewage treatment plant, for example, can be rich in nutrients. Also, some food processing operations where beer, tomato paste, milk, sugar, etc. enter the cooling system can have severe corrosion and biological problems. Another potential lies with air quality. Cooling towers located near bakeries, for example, show an increased tendency to grow biological material due to the molds and yeast. Biological concerns run the gamut from nearly zero to very substantial. Whatever the case, an appropriate solution must be developed. The traditional approach is for the operator to alternate between two liquid biocides adding them at a predetermined frequency. Two different formulations are often used to avoid an immunity being developed to just one. Unlike scale and corrosion chemicals that are metered into the system frequently, biocides are typically administered every few days to 'shock' the system. Other chemicals for biological control include chlorine, iodine, bromine and ozone. These are not rotated with other chemicals; They are fed by themselves. Ozone is also used to prevent scale. Be certain to specify Viton pump seals when using ozone; Standard seals will fail quickly.

The cooling tower has the misfortune of being a handy receptacle for the addition of water treatment chemicals which are almost always corrosive in concentrated form. The point of injection of scale and corrosion chemicals is important. They

should not be dripped into the top of a cooling tower where the air can blow them against metal components. They should be introduced into the piping, where they will disperse quickly. Similarly, pelletized chlorine tablets tossed into the sump can burn holes through the basin floor.

There is also a general failure to recognize that cooling water quality can be very dynamic. Do not, for example, make the mistake of installing a new tower, placing it into operation, and ignoring the water treatment for a few days. Some closely coupled systems with small water volumes (evaporative condensers and fluid coolers lending the best examples) can be scaled in a matter of hours.

Cooling towers do not suffer from the accumulation of scale as quickly as their evaporative condenser and fluid cooler counterparts. Scale can be allowed to remain in a cooling tower if it is not so thick as to inhibit airflow. Just descale the device being cooled- typically a condenser bundle- by chemical or mechanical means. When cleaning chemically, isolate the heat exchanger and circulate the chemical solution through it with an auxiliary pump following manufacturer recommendations. Evaporative condensers and fluid coolers have been successfully descaled using a five percent (by weight) solution of inhibited sulfamic acid. It must be monitored very carefully- ask the manufacturer for recommendations.

Filters are an additional water treatment option, opten used with chemical treatment. A 100 ton cooling tower processes about 40 tons of air in an eight hour period retaining all the air borne debris in the tower water. Ideally, this debris would stay in suspension and be removed by the bleed but a good portion typically manages to settle in the tower basin and on heat exchange surfaces. Such debris tends to reduce the effectiveness of water treatment chemicals. Full flow filtration is generally limited to devices that operate with low pressure drop and those that have an ability to purge debris while in operation. Strainers that swing into the flow stream while others backwash and centrifugal separators can be used full flow. Another option is a centrifugal separator, which directs the water flow tangentially into a cylinder causing the water to rotate.

Debris particles that are heavier than water migrate to the outer wall area and slither down into a collection bowl that is periodically blown down. Very little water is lost. Typical performance is 97.8% of solids with specific gravity exceeding 1.2 down to 45 microns. Side stream filtration is more common in cooling tower applications. The scheme uses a small dedicated pump to draw dirty water from the sump, direct it through a filtration device and sent it back to the basin.

In addition to being smaller and less expensive, side stream filtration has the advantage of allowing the return water to be routed through a perforated PVC distribution pipework or- better yet- nozzle jets that agitate the water at the basin floor placing the debris in suspension and increasing its chances of being drawn into the filter suction piping. The actual side stream filtration device can be a centrifugal separator, strainer device or sand bed filter. The sand bed filter is especially effective in removing particulate matter- even light weight biological material killed

by biocides. Filtration to ten microns is commonplace. When the sand begins to get clogged, a pressure differential switch signals a backwash cycle that lasts about three minutes. The valves reposition to reverse the flow through the sand bed lifting it and carrying off debris to the drain. Side stream filtration devices applied to open cooling towers are typically sized to pass the entire system volume at least once each hour for good water clarity. A second filter selection method can be used when the system volume isn't known- and on all evaporative condensers and fluid coolers- that relates the filter size to the amount of air ingested:

$$\text{Filter GPM} = \text{Cooling Tower CFM} \times .0004$$

Use both methods when possible and use the largest as a starting point. Then, apply a judgment factor increasing the size to accommodate blowing dust or sand. One complaint water treatment personnel have with filters is they discharge system chemicals during backwash and therefore prefer backwash be accomplished with city water. The city water backwash example is actually rare. It is far more common to employ system water for backwash. City water backwash is really only necessary when sumps have insufficient volume to keep from running out of water during the backwash cycle. From the filter supplier's side, city water pressure can be too high or too low causing the backwash cycle to be too vigorous or too lethargic. They feel more comfortable with the known pressure generated with their own pump delivering 'system' water for backwash. Filter backwash doesn't diminish the residual chemical level appreciably when system volumes are large; However, closely coupled systems with 'small basin' cooling towers can suffer severe chemical depletion from backwash. There should be no reason why controls can't be provided and set to prevent backwash until it is appropriate to bleed and to feed the appropriate chemical dosage after bleeding.

GLOSSARY OF TERMS

There are a number of terms with rather rigorous definitions within the context of cooling tower technology. It is worthwhile to learn these definitions.

Acceptance Testing: Test procedures to determine the water cooling capacity of towers. Instrumentation used and measurement procedures should be those recommended by the Cooling Tower Institute (CTI) in its "Acceptant Text Procedures. "

Air Inlet: That portion of the cooling tower structure in which air is drawn into the system.

Ambient Dry-Bulb Temperature: External outdoor temperature as indicated by a dry-bulb thermometer and expressed in degrees Fahrenheit.

Ambient Wet-Bulb Temperature: The temperature in degrees Fahrenheit to which air can be cooled, making it adiabatic to saturation by the addition of water vapor. In practical terms, the wet-bulb temperature is the temperature indicated by a thermometer, the bulb of which is kept moist by a wick and over which air is circulated.

Approach or Approach to the Wet-Bulb: The difference in temperature (°F) of the cold water leaving the tower and the wet-bulb temperature of the ambient air.

Balancing Valve: Hand or mechanically operated valve installed in each riser pipe of a multicell tower to control water flow.

Basin: The area at the bottom of the tower for collecting cold water. Crossflow towers have a hot water distribution basin at the top and, in some cases, a water basin between the top and bottom basins.

Blowdown (Purge): The continuous or intermittent wasting of small amounts of circulating water. Its purpose is to prevent an increase in the concentration of solids in the water due to evaporation, normally expressed as a percentage of the water being circulated.

Capacity: The average amount of water circulating in the cooling system at any given time, expressed in gallons per minute.

Casing: The vertical enclosing side- or endwall of a tower, exclusive of the air inlet louvers.

Cell: A unit consisting of a distribution system, mechanical equipment and partition walls. A single tower can have several independent cells. Individual cells can be shut down, or several cells can be run on partial capacity.

Cellular Film: Asbestos fill packing that converts water droplets into a thin molecular filter for more efficient cooling. Less static pressure is encountered than with splash-bar fill, thus permitting the use of higher air velocities.

Concentration Cycles: Comparison of dissolved solids in makeup water with solids concentration in the circulating water.

Cooling Factor: The ratio of the pounds of water circulated per unit of time to the pounds of dry air cooling the water per unit of time.

Cooling Tower Institute: International organization of cooling tower engineers, manufacturers and users, dedicated to improving the professional and technical state of the art.

Counterflow: A system in which air encounters the hot water at a 180° angle. Air enters near the base of the tower and moves upwards through the fill and falling water.

Crossflow: A system in which air encounters the hot water at a 90° angle. Air enters through the entire sidewall and moves horizontally through the fill and water.

Delta Temperature (Range): Difference between entering and leaving water temperatures.

Design Conditions: Thermal parameters for which the cooling tower is purchased. They consist of a given gpm flow of water entering the tower at a specific temperature, cooling through a given range, leaving the tower at the required temperature, and having a designated approach to a stated wet-bulb temperature.

Diffusion (Redistribution) Deck: A device below the hot water distribution basin of a crossflow tower to break up the water going through the orifices before it goes through the fill.

Distribution System: Mechanical method of passing hot water over the fill uniformly. Low-pressure spray-through piping and nozzles are usually used in counterflow towers; gravity drop is normally utilized in crossflow towers.

Drift: Entrained water droplets that escape from the tower with the exhaust air expressed as a percentage of water circulated.

Drift Eliminator: Baffling that causes discharging hot air containing entraining water droplets to change direction a number of times. Droplets hit the eliminator surface and fail back into the tower.

Fill Packing: Specially designed baffling used to provide a large surface area for heat transfer. Two classes of materials are used: splash bars of wood, metal transite or plastic and film pack (cellular fill). The splash type cools the water as the droplets bounce down a series of bars in the air stream; film packing converts droplets into a thin film.

Fog: A mist formed where the ambient air cannot absorb all the plume's moisture. The intensity of the fog is a function of the heat rise of air passing through the tower and the temperature and humidity of the ambient air. Fog plumes are normally permissible since there are no droplets of water raining out of the discharge area; however, fog may cause icing of nearby roads and may restrict visibility.

Forced Draft: Air introduced at the bottom of the tower is forced to the top by a centrifugal blower.

Heat Load: Amount of heat (in Btu) dissipated in a cooling tower. It is equal to the weight of water circulated per unit of time multiplied by the cooling range.

Induced Draft: Air mover, usually an aerial fan, on top of the tower pulls air up through the fill and out the stack.

Latent Heat of Vaporization: The heat required to change a liquid into a vapor without a change in the temperature or pressure.

Louvers: Baffles used for changing the direction of air flow into the tower in a uniform, parallel manner. They are also used in preventing water droplets from

splashing out of the tower as they fall through the structure. The same principles that apply to controlling air flow with fans applies here.

Makeup: This term refers to the water required to replace the circulating water that is lost by evaporation, drift, blowdown and leakage. It is expressed as a percentage of the water circulated and normally is automatically controlled by a float valve.

Net Effective Volume: A portion of the total structural volume in which the circulating water is in intimate contact with the air flowing through the tower (expressed in cubic feet).

Performance: The measure of the tower's ability to cool water. It is usually expressed in terms of cooling a quantity of water (gpm) from a specified hot water temperature to a specified cold water temperature at a stated wet-bulb temperature.

Performance Curve: A graphic representation of the relationship of water temperature, approach, wet-bulb temperature, range, static pressure and air movement.

Plenum: An enclosed chamber in which pressure is higher than atmospheric pressure.

Plume: Visible manifestation of water vapor condensing as warm, moist air mixing with the cooler, outside air.

Psychrometer: Instrument used to measure the wet-bulb temperature.

Pumping Head: The energy required to raise water to the distribution elevation and overcome friction losses through pipe, valves, fittings and nozzles. It is expressed in feet of liquid the pump must move and is equal to the total friction loss, static head and pressure drop through the distribution system.

Range: The numerical difference between the temperature of the hot water entering the cooling tower at the distribution system and the temperature of the colder water leaving the sump basin.

Recirculation (Recycle): Hot exhaust air forced downward and back into the cooling tower raises the wet-bulb temperature of the entering air above dry-bulb temperature, impairing tower performance. It is usually caused by design, wind or placement problems.

Sensible Heat: The heat required to change the temperature of air or water.

Static Pressure Drop: The reduction of air movement through the tower resulting from resistance of internal components such as air-intake louvers. Oil packing, water distribution system, internal supporting beams, drift eliminators and fan stack configuration.

Sump: Depressed section of the cold water collecting basin, from which cooled water returns to the heat source.

TDS: Total dissolved solids contained in solution in the cooling water system.

Water Load: Circulating rate of water over the tower, expressed in gallons per minute.

Wet-Bulb Temperature: The temperature of saturated air. The lower the wet-bulb temperature, the more exchange of heat a cooling tower can do. A tower cannot cool the water to a temperature below the wet-bulb temperature of the entering air.

Windage: The loss of water through the air-intake louvers as a result of malfunctioning of the wind check walls in the lower section of the tower.

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Chapter 3
EVAPORATING AND DRYING EQUIPMENT

INTRODUCTION

Evaporation and drying are standard unit operations. Evaporation is used in such applications as solvent recovery, product preparation, recovery of valuable by-products, in waste processing. Drying is a separate and distinct unit operation, but can be coupled with evaporative techniques depending upon the application. Drying can be used in similar applications to evaporation, but more often as a principle unit operation aimed at product finishing and waste processing. Specific examples and equipment configurations are discussed in this chapter. Often equipment designs are very unique to the application, and therefore the specifier must have a keen understanding of the nature of the operation and the process materials handled.

EVAPORATORS

Evaporators are used extensively throughout the chemical process industries for applications ranging from concentrating black liquors for recovery furnaces in paper manufacturing, to various product drying operations in the food industry, to the manufacturing of a wide range of semi-dry and low moisture content chemical feedstock products and intermediates. Refer to Figure 1 for a simple classification system of evaporating equipment. Concentrating a solution by the evaporation of part of the solvent is a widely practiced. When the solvent is water, the function of the evaporator is to vaporize part of the water, resulting in a concentrated solution. The vapor is of no use except, perhaps, for its heat value, and it is the residual solution that is saved. This is the distinction in general terms between evaporators and a still. In the latter it is the vapor that has value, while generally, though not always, the residual liquor does not. Heating is usually accomplished with steam at low pressure. Typically saturated steam is used without superheat. Superheat does not furnish sufficient heat units to be of importance (e.g., total heat content of saturated steam at 40 lb gauge is 1169.4 Btu/lb, of which 933.3 Btu is latent heat, the portion generally utilized); while 40 lb gauge pressure steam with 100' of superheat has a total heat content of 1218.4 Btu; hence, this adds 47 Btu additional to latent heat, which is the same as for saturated steam. This heat is known as the latent heat of steam, also the heat of condensation, that vaporization is delegated to.
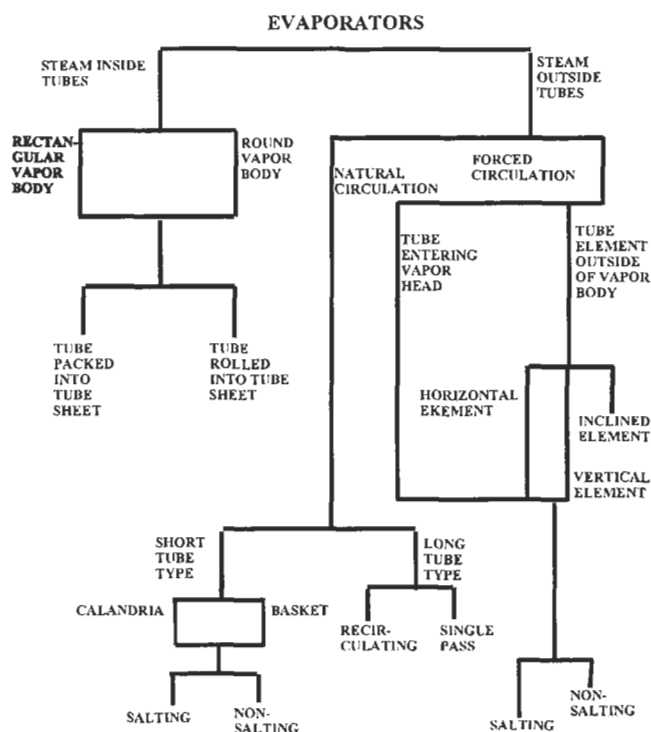
Figure 1. *Classification system for evaporating equipment.*

Major evaporator designs include forced-circulation, long-tube vertical (both rising and falling film), and calandria-type evaporators. The economics of a particular process will dictate the evaporator style and model best suited to a particular application. Forced-circulation and calandria evaporators are required for processes where crystals are formed. These evaporators are designed to keep crystals suspended in solution to prevent scaling of the equipment. Long-tube vertical evaporators are used to concentrate a liquid that does not have solids present.

Evaporators require a source of heat to operate. This heat may be supplied from a boiler, gas turbine, vapor compressor, other evaporator, or a combination of sources. Multiple effect evaporators are very popular when cheap, high pressure steam is available to heat the system. A Mechanical Vapor Recompression evaporator would use electricity or a gas turbine to drive a compressor that recycles the heat in the evaporator.

Evaporators have performed successfully in a number of industrial applications. Typical materials that are processed in evaporators include Caustic Soda, Caustic Potash, Sodium Carbonate, Sodium Dichromate, Sodium Nitrate, Ammonium Nitrate, Phosphoric Acid & Superacid, Potash, Urea,   Glue,   Glycerine,

Tankwater, Bright dip acid (phosphoric), Cyanide rinse bath, Pickle Liquor, Sodium Aluminate Liquor, N.S.S.C. Liquor, Kraft Liquor, Soda Liquor, Sulfite Liquor, Stillage, Corn Syrup, Gelatin, Salt, Soybean Oil, Steepwater, Sugar, Whey, Mercerizing Caustic, Nylon Salt, Rayon Spin Bath, and Sodium Sulfate.

As the name implies, the liquor in a Forced-Circulation Evaporator is pumped through the tubes to minimize tube scaling or salting when precipitates are formed during evaporation. A forced-circulation evaporator (with a submerged inlet) complete consists of a single-pass vertical heat exchanger, elutriating leg, cyclone, and top mounted barometric condenser. Slurry is pumped from the bottom cone of the vapor body through the tubes of the vertical heat exchanger, where heat is added, and back into the vapor body where evaporation occurs. Sufficient slurry height (submergence) is maintained above the tangential inlet on the vapor body and above the top tubesheet of the heat exchanger to suppress mass boiling in the inlet and prevent surface (local) boiling on the tube surface. This is necessary to preclude salt precipitation on the tangential inlet and tubes.

A high circulation rate is provided for adequate tube velocity to achieve good heat transfer. Therefore, lower slurry temperature rises are assured which minimize supersaturation of the solution. A sufficient quantity of salt crystals is suspended in the circulating system to provide seed crystals in the boiling zone for salt growth. Adherence to these basic principles of crystallization results in coarse crystals and minimal wall and tube salting, so less equipment washing is required. This conserves energy because less steam is required to boil wash water and this increases on-stream time for the evaporator. The circulating pump is usually of the axial-flow, single-elbow design, well-suited for the high flow rates and low pressure drops. These heavy duty pumps operate at low speeds, which reduce maintenance and minimize mechanical attrition of the salt crystals. Circulating piping interconnects the vapor body, the heat exchanger and the pump. Conical liquor chambers provide gradual, low pressure drop transitions from the circulating piping to the tube bundle which is particularly important for the establishment of uniform feed to the tubes. To provide for thermal expansion without expansion joints, the circulating pump base is spring-mounted. As an alternative, the entire pump may be hung from the circulating piping. The tangential inlet provides excellent mixing of slurry in the vapor body because of the circular motion it creates. Secondary vertical currents are also generated, mixing body slurry with the hotter slurry entering the vapor body to reduce the degrees of flash. This agitation minimizes salt buildup on the bottom cone of the vapor body. A swirl breaker is provided in the circulating slurry outlet. The vapor body is conservatively designed both in diameter and height. It is important to have an adequate free space above the liquor level to allow the liquor droplets entrained in the vapor leaving the boiling surface to reach equilibrium and return by gravity to the circulating slurry. The large diameters result in low vapor velocities which minimize entrainment and provide adequate retention time for salt growth. A mesh-type entrainment separator may be

installed in the upper portion of the vapor body to reduce solids carryover to normally less than 50 parts per million parts of vapor. Other types of entrainment separators are also available.

The elutriating leg, attached to the bottom cone of the vapor body, is a convenient device for thickening the slurry it receives from the vapor body and for salt crystal washing and classification. Slurry enters the top of the leg through a unique slurry-inlet device, which improves washing efficiency by reducing agitation in the leg. Salt crystals are fluidized and washed in the leg with a portion of the feed liquor which enters the bottom cone and is distributed with a perforated, dished head. Smaller crystals are washed into the vapor body for additional growth and the larger crystals are discharged from a connection near the bottom of the leg.

A low-pressure-drop liquid cyclone is sometimes used to clarify liquor discharged from the evaporator. The driving force is the pressure drop across the circulating pump. Thickened slurry is returned through a wide-open cyclone underflow connection to the circulating piping before the pump suction.

An innovation is a direct-contact condenser mounted on the vapor body. A short piece of vertical pipe connects the vapor body with the condenser to minimize piping and pressure drop. This design also eliminates structural steel for support of a separate condenser. For cooling tower applications, the hotwell is elevated to permit gravity flow of water from the hotwell to the top of the cooling tower, thus eliminating the need for a pump.

A forced-circulation evaporator with horizontal heat exchanger and a top-mounted stripping column operates in the following manner. Reflux liquid is introduced on the top tray of the column to strip one or more compounds from the water vapor. Stripping columns are used for special applications and are provided either integral with the evaporator or as a separate column. The columns are for the recovery of valuable components from the water vapor and for the reduction of volatile pollutants. Stripping columns are usually supplied with ballast and valve trays for caprolactam and boric acid service. Spray columns have been used for the recovery of fluorine generated along with the vapor during the concentration of phosphoric acid.

Heat exchangers can be located on the discharge or suction side of the circulating pump. In some cases, both discharge and suction-side heat exchangers have been provided in the same circulating system to maximize heat-transfer surface. Multiple circulating systems are provided for the same vapor body on large evaporators. Well designed systems include special provisions to reduce tube plugging caused by salt lumps.

A variety of forced-circulation evaporator designs are available, with and without elutriating legs or cyclones. Different inlets and outlets are used on the vapor body, tailored for the particular application. For some applications, such as super-

phosphoric acid and red liquor, it is necessary to use unsubmerged-inlet evaporators to reduce the volume in the system to a minimum or to minimize foaming. Because of the higher head created by the unsubmerged inlet, it is sometimes necessary to use a mixed-flow circulating pump instead of an axial-flow pump.

Typical Configurations

A standard form of evaporator is an upright cylinder, within which a nest of upright steam tubes is placed. The horizontal steam tubes are also built into the upright cylindrical body. Instead of tubes, suitably placed coils, within which steam circulates, are installed for small-scale and batch operations. Certain evaporators have both tubes and (at a lower level) coils. The function of an evaporator is to transfer heat, mainly the latent heat of the steam from the steam side through the metal wall of the vessel to the liquor on the outer side. The resistance to the passage of the heat is not only that of the metal, which is indeed very small, which means that the heat transfer coefficient is high, but also that of the condensed steam film on the one side and a film of liquor on the other side. The condensed steam film, which is essentially hot water, has a moderate resistance to heat, partly because its thickness is low; hence, its coefficient of transfer is 2,000 or 3,000. Figures 2 through 8 provide examples of common equipment that will assist the reader in becoming familiar with the various configurations and operational modes for evaporating equipment.
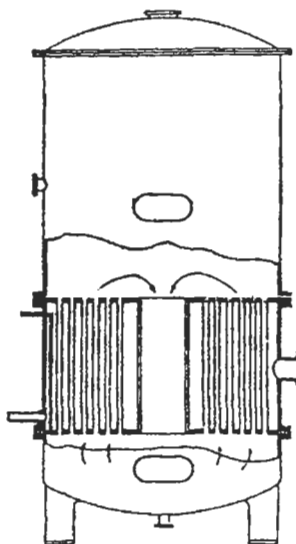
Figure 2. *Vertical evaporator.*   Figure 3. *Vertical long-tube evaporator.*
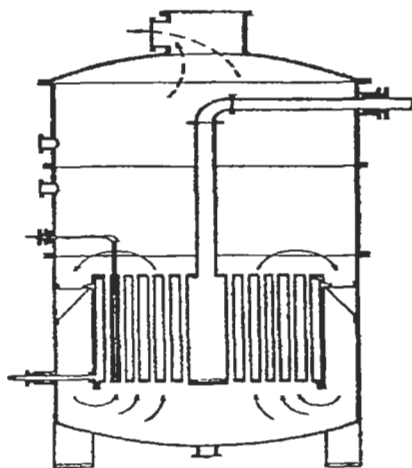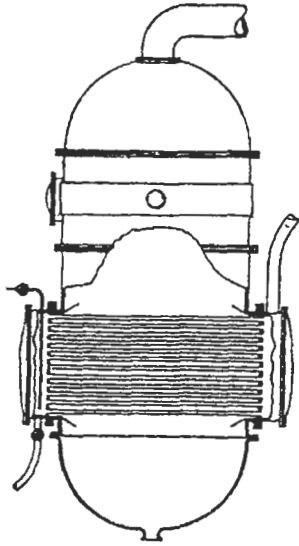
Figure 4. *Horizontal design.*
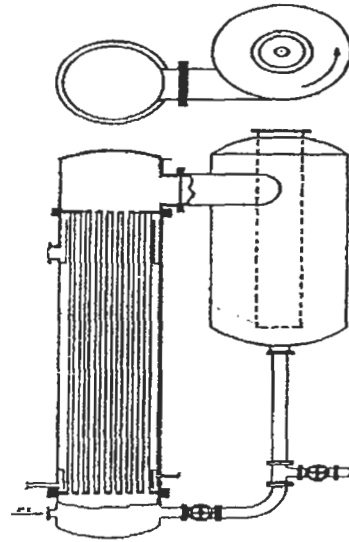
Figure 5. *Natural circulation, long tube film evaporator.*
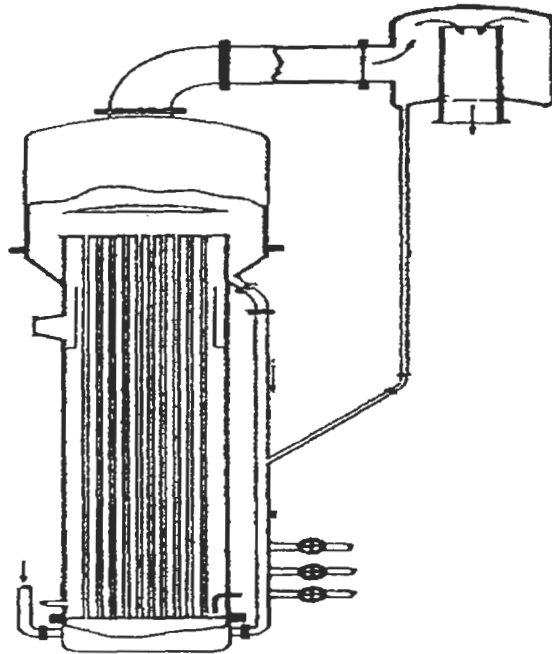
Figure 6. *Natural circulation, long tube-vertical film evaporator.*
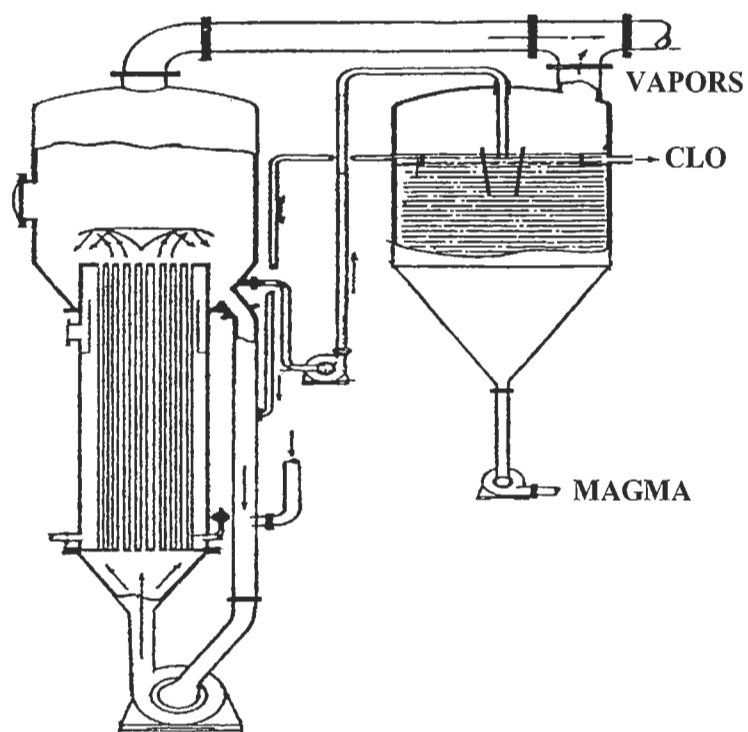
Figure 7. *Forced circulation evaporator.*
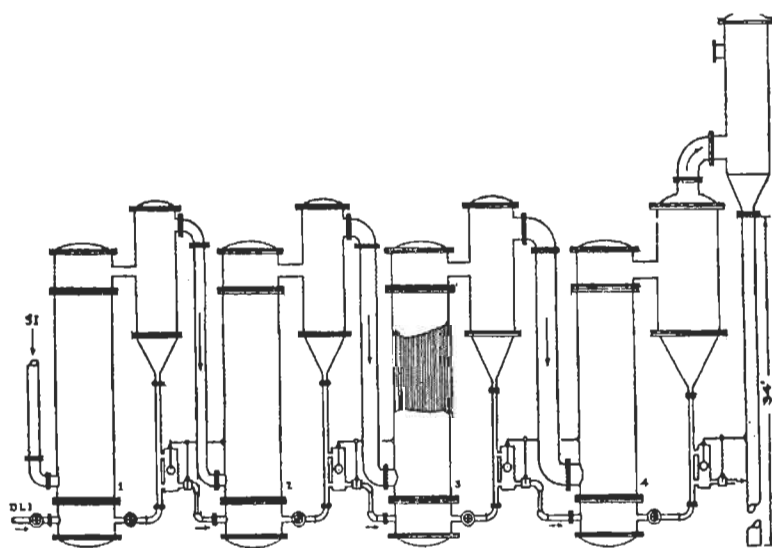
Figure 8. *Quadruple effect, natural circulation vertical tube evaporator.*

A simplified example of a Forced-Circulation Type Evaporator is illustrated in Figure 9 (in this figure the letter designations are: A. Heating element shell; B. Vapor head; C. Downtake pipe; D. Circulating pump; E. Deflector; F. Steam baffle; G. Non-condensed gas outlet; H. Condensate outlet; J. Salt settler; K. Liquor level indicator). The tubes are longer and narrower than in the standard vertical type and are contained in a heating element A between two tube sheets. This tube bundle projects part way into the vapor head B. A return pipe C leads from the vapor head to the inlet of a pump D and this pump delivers the liquid to the tubes with a positive velocity. As the liquid rises through the tubes, it becomes heated and begins to boil, so that vapor and liquid issue from the tops of the tubes at a high velocity. This material strikes against a curved deflector E that throws the liquid downward in a sheet or curtain and makes an effective separation of liquid and vapor. The steam connection is near the bottom of the tube bundle.

Just inside the shell of the tube bundle is a cylindrical baffle F that extends nearly to the top of the heating element. The steam rises between this baffle and the wall of the heating element and then flows downward around the tubes. This displaces non-condensed gases to the bottom, where they are removed at G. Condensate is removed from the bottom of the heating element at H. This evaporator is especially suited for foamy liquids, for viscous liquids, and for those liquids which tend to deposit scale or crystals on the heating surfaces. Vessel J is a salt separator.
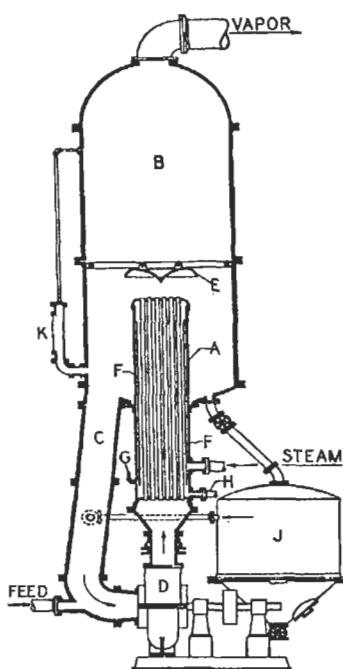
Figure 9. *Forced circulation evaporator.*

Basket Type Evaporators - the first basket - type evaporator was built in 1877 and an example of this type is shown in Figure 10. In this evaporator the liquid is inside the tubes, the steam is outside the tubes, but the downstage instead of being central is annular. In this construction the entire heating element is a single unit which may be removed bodily for repairs. The evaporator has a conical bottom, but flat bottoms are also common in this type.

The tube proportions in the basket evaporator are about the same as in the standard vertical. One important feature of the basket-type evaporator is the ease with which a deflector may be added in order to reduce entrainment from spouting. The boiling in the vertical-tube evaporator is quite violent, and this tends to cause entrainment losses. This condition is accentuated if the liquor level in the evaporator is low. A baffle such as shown in Figure 9 largely prevents these losses and is much more easily added to the basket type than to the standard type. Other differences between the standard and the basket type are largely details of construction.
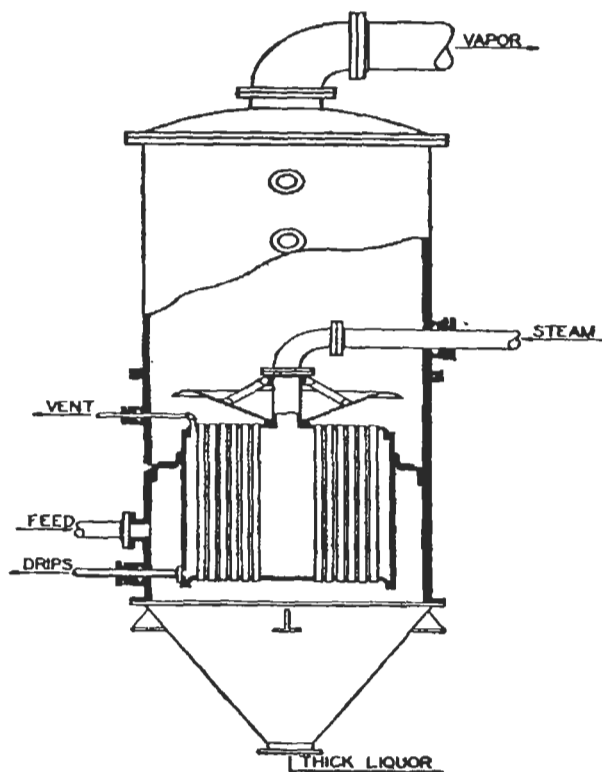
Figure 10. *Basket type vertical tube evaporator.*

Evaporators, Vertical - Tube Type - An example is shown in Figure 11. This evaporator is characterized by tube sheets A extending across the body, and a central downstage B. Tubes are rolled between these two tube sheets, and steam is introduced as shown, so that the liquor is inside the tubes and the steam is outside them. As the liquor boils, it spouts up through the tubes and returns through the central downstage. Condensate is removed from any convenient place on the bottom tube sheet such as C, and non-condensed gases are usually vented from somewhere near the upper tube sheet as at D. The exact position of feed E and discharge F is variable. There are many variations of this design; a conical bottom may be used instead of a flat bottom; and the proportions of tube length to tube diameter, ratios of body height to tube length, and other details are often modified. The first vertical - tube evaporators were built without a downtake. These were never satisfactory and the central downstage appeared very early. There have been many changes over the years, such as downtakes of different cross-section, downtakes located eccentrically, a number of scattered downtakes instead of one central one, downstage pipes entirely external to the body of the evaporator, and others.
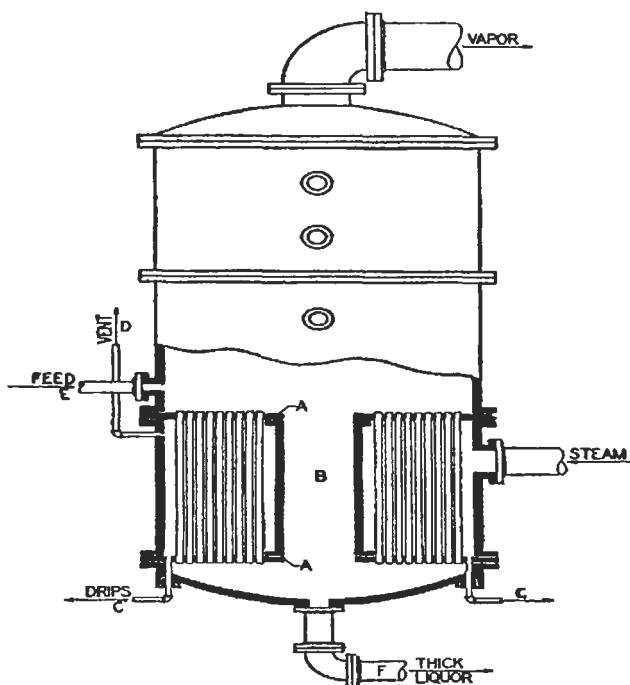
Figure 11. *Vertical tube evaporator: (A) Tube sheets; (B) Downtake; (C) Condensate outlet; (D) Non-condensed gas outlet; (E) Liquor inlet; (F) Thick liquor outlet.*

Evaporators, Horizontal-Tube Type - The basic horizontal-tube evaporator is illustrated in Figure 12. The body of this evaporator is the liquor compartment and is in the form of a vertical cylinder. It is closed, top and bottom, with dished heads, although the bottom may be conical. The lower body ring is provided on opposite sides with steam compartments, closed on the outside by cover plates and on the inside by tube sheets. Between these tube sheets are fastened a number of horizontal tubes. The two steam chests with their connecting tubes form the steam compartment, and the tube wall heating surface. Steam is introduced into one steam chest and as it flows through the tubes it washes non-condensed gases and condensate ahead of it, so that these are withdrawn from the opposite steam chest.

In ordinary operation only condensate and non-condensed gases are removed from the exit steam chest. The connection for feeding the liquid to be evaporated may be attached to the body at any convenient point (D), but the discharge for thick liquor is usually in the center of the bottom (E). Suitable brackets are cast on the bottom to rest on the supporting steel. Most evaporators are provided with sight glasses.
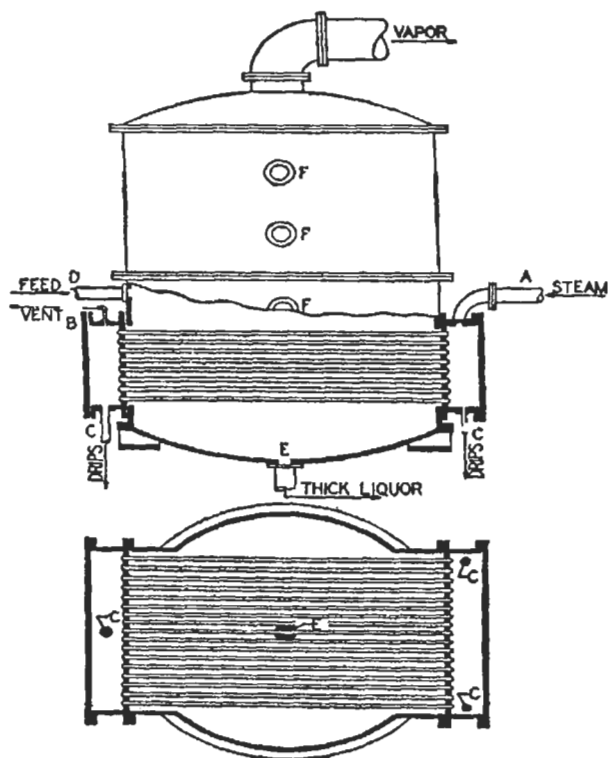
Figure 12. *Horizontal tube evaporator: (A) Steam inlet; (B) Vent for non-condensed gas; (C) Condensate outlet; (D) Liquor inlet; (E) Liquor outlet; (F) Sight glass; (G) Vapor outlet.*

There are many modifications of the horizontal-tube evaporator, but these consist largely of changes in the shape of the body castings and not at all in the general arrangement or interrelationship of the parts. The horizontal-tube evaporator is best suited for non-viscous solutions that do not deposit scale or crystals on evaporation. Its first cost per square foot of heating surface is usually less than that of the other types of evaporators.

A common design involves steam outside the tube which is shown in Figure 13. This evaporator consists of a cylindrical shell A closed by tube sheets B at either end. The shell may be from 8 to 20 ft. long. A number of tubes are fastened in these tube sheets by rolling. This term refers to a method of fastening a tube in a tube sheet by means of a tool containing rollers and inserted into the end of the tube. The rollers are so mounted that as the tool is rotated the tube is gradually expanded until it makes a tight fit with the tube sheet. Over these tube sheets are fitted distributing plates which have return bends cast in them in such a way that the tubes are connected in series in groups of three or five. One of these distribution plates carries feed connections that register with the end of the first tube of each group. The other distribution plate has openings opposite the other end of each group. Around this end is built a large separating chamber D provided with baffles E. The shell is provided with an inlet connection for steam F and outlets for noncondensed gases at H and condensate at G.
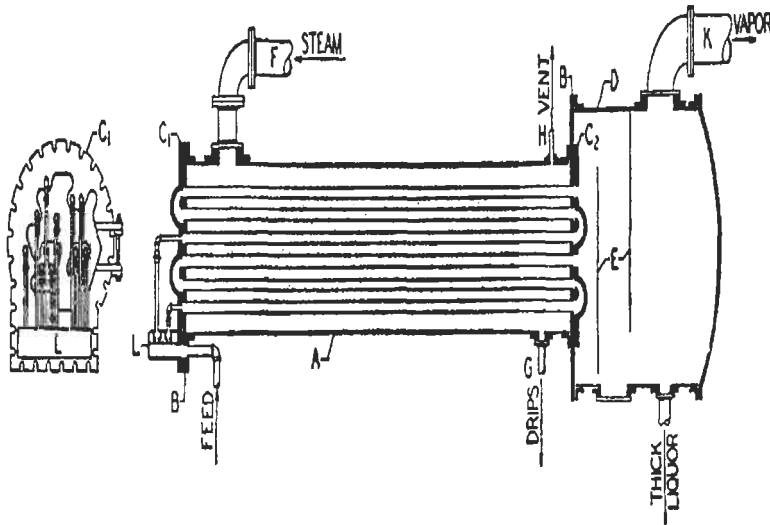
Figure 13. *Steam outside tube evaporator: (A) Shell; (B) Tube sheets: (C₁, C₂) Distributing plates; (D) Vapor head; (E) Baffles; (F) Steam inlet; (G) Condensate outlet; (H) Non-condensed gas vent; (J) Thick liquor outlet; (K) Vapor outlet; (L) Liquor feed box.*

As the liquid is introduced into the feed end of each group of tubes, it begins to boil, and the volume of vapor so formed is extremely large as compared to the volume of liquid from which it is formed. As the liquid passes down the tube, therefore, the combined volume of steam and liquid becomes larger, the velocity is correspondingly greater, and a mixture of steam and spray issues from the end of each coil and strikes against the baffles that serve to separate vapor from liquid. The concentrated liquid is drawn off at the bottom of the separating head at J, and the vapor is removed from the top at K. The liquor is not returned from J to the feed end but passes through the coils only once. All of the feed pipes are usually connected to a common feed box L, which is cast as a part of the lower edge of the feed-distributing plate. The individual feed lines extending from this box to the various coils contain throttle valves or orifices to distribute the feed evenly over the various coils.

Solvent evaporators are a workhorse in the CPI. In a single-effect, LTV rising-film evaporator evaporation occurs inside the evaporator tubes, so it is used primarily to concentrate non-salting liquors. To provide for good heat-transfer rates, the Delta T (i.e., temperature difference) between the heat-transfer medium and the liquor should be greater than 15 °F, and preferably greater than 20 °F. Tubes are normally 3/4" to 2" in diameter and from 10 to 30 feet long. Operation of the rising-film evaporator is straightforward. Liquor is fed into the bottom liquor chamber and then into the tubes. There it is heated with condensing steam or any other suitable heat-transfer medium, such as Dowtherm or hot liquor. If the vapor pressure of the feed equals or exceeds the system pressure at the bottom tubesheet, vaporization will occur immediately. For colder feed, the lower portion of the tubes is used to preheat the liquor to its boiling point. Vaporization then begins at that height within the tubes where the vapor pressure of the feed liquor equals the system pressure. As the liquor climbs up the inside of the tubes, additional vapor is generated and the velocity of the liquid-vapor mixture increases to a maximum at the tube exit. The outlet mixture impinges upon a deflector, mounted above the top tubesheet of the heat exchanger, where gross, initial separation of the liquid from the vapor occurs. Additional liquor is separated from the vapor by gravity as the vapor rises in the vapor body. A mesh-type or centrifugal entrainment separator can be installed near the top of the vapor body to remove most of the remaining traces of liquid from the vapor. The exit vapor is conducted to the next effect of a multiple-effect evaporator, to a compressor or to a condenser.

Heat-transfer rates are enhanced in the non-boiling section by surface or local boiling and in the boiling section by nucleate boiling. As expected, the heat-transfer rates in the boiling zone are several times greater than those in the non-boiling zone, so it is important to reduce the non-boiling zone to a minimum.
Different two-phase flow schemes are created in the boiling zone, including *slug flow*, where a slug of liquor is followed by a slug of vapor, similar to the perking in a coffee percolator; *annular flow*, where a ring of liquor encases a center core

of vapor and liquid mist; and *mist flow*, where vapor blankets the tube surface. Mist flow should be avoided because poor heat transfer results when there is not enough liquid present to wet the tube walls. To avoid mist flow, it is sometimes necessary to recycle concentrated product from the vapor body to the bottom liquor chamber so as to supplement the feed liquor.The LTV rising-film evaporator can also be adapted to concentrate moderate scaling liquors when a pump is used to recycle concentrated product from the vapor body to the bottom liquor chamber. This is known as forced recirculation. The vapor body is integral with the heat exchanger. When the heat exchanger is too large to use the integral configuration, when quicker access to the tubes is desired for maintenance, or when surge volume in the vapor body is required for level control, the vapor body is separated from the heat exchanger. A skirt-type baffle replaces the deflector as the initial separator.

In a falling-film evaporator with separate vapor body and heat exchanger, liquor is fed into the top liquor chamber of the heat exchanger where it is distributed to each tube. The liquor accelerates in velocity as it descends inside the tubes because of the gravity and drag of the vapor generated by boiling. Liquid is separated from the vapor in the bottom liquor chamber of the heat exchanger and with a skirt-type baffle in the vapor body. A supplemental entrainment separator can be installed in the upper portion of the vapor body to reduce liquid entrained with the vapor to a minimum. A direct-contact condenser is used to condense the vapor with water. Concentrated liquor is discharged from the bottom liquor chamber and cone bottom of the vapor body. The vapor body can also be provided as an integral component of the heat exchanger, except the heat exchanger would be located above the vapor body in the falling-film configuration. Evaporation also occurs inside the tubes of the falling-film evaporator. The unit can be used to concentrate the same non-salting liquids concentrated in rising-film evaporators, and it is suitable for concentrating more viscous liquors. Tube sizes and lengths are comparable to those for rising-film evaporators. The falling-film evaporator is particularly useful in applications where the driving force in temperature difference between the heat-transfer medium and the liquid is small (Delta-T's of less than 15 ^{0}F). The retention time for liquor in this evaporator is less than that for a rising-film evaporator. The combination of short liquid retention time and the ability to operate at a low Delta-T makes the falling-film evaporator ideal for concentrating the most heat-sensitive materials.

High heat-transfer coefficients are attained in falling-film evaporators when a continuous film of liquid, preferably at its boiling point, flows down the inside tube wall with a vapor core in the tube center. For some applications, however, it is necessary to supplement an insufficient quantity of feed liquor with product liquor pumped to the top liquor chamber to avoid vapor blanketing of the inside tube surface.

The *Calandria Evaporator* is applied less often today than it was years ago. For most applications, however, the lower equipment costs for other designs has prompted the replacement of calandria evaporators with rising-film, LTV falling-

film and forced-circulation evaporators. The *Calandria Evaporator* has a heat exchanger (with tubes usually less than six feet long) integral with the vapor body. The level is maintained in the upper portion of the tubes or above the top tubesheet and the circulation pattern is up through the tubes and down through a central pipe called a "downcomer". Circulation is created by the difference in specific gravity between the body liquor and the heated liquor and vapor generated inside the tubes, plus a vapor lift effect. The *Calandria Evaporator* can be used for salting-type applications; however, an agitator located inside or beneath the downcomer is recommended to suspend the salt crystals in the lower portion of the body. Although the agitator creates some flow through the tubes, most of the flow is still created by "thermo-syphon". The calandria is also used for batch-type concentration of liquors.

Applications and Process Schemes

There are numerous applications in solvent recovery processes where evaporation equipment are employed. Figure 14 provides an example of a process scheme for toluene-di-isocyanate recovery. This is an example of continuous vacuum evaporation of distillation residues.
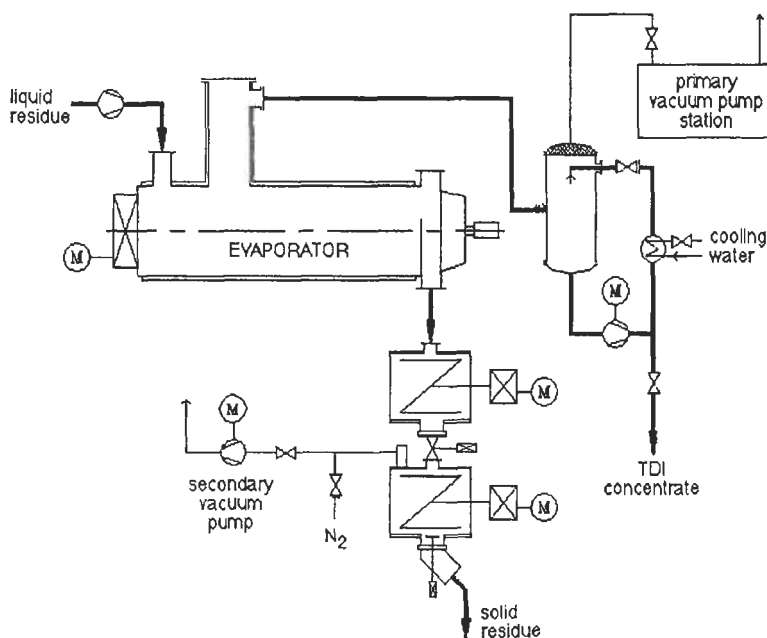
Figure 14. *Process flow scheme for TDI recovery by evaporation.*

The vaporized solvent is recovered by condensation. The remaining non-toxic waste typically is free-flowing powder with residual solvent contents that are below 0.5 %. This is an economical process scheme involving only a single-step process for the complete recovery of the solvent. The design is a closed system, which makes it environmentally safe, and yields a non-toxic solid residue for disposal. Other applications for the process configuration illustrated in Figure 14 include handling pasty/sticky residue with toxic-high boiling solvents from chemical or pharmaceutical productions, and the preparation of washing solutions.

Another example of a solvent recovery process is that of DMAC (Dimethylacetamide) recovery, illustrated in Figure 15. In this example, the solvent recovery involves continuous evaporation under vacuum. The vaporized solvent exits through a dust filter and is recovered for reuse by condensation. The dried residue can be either a free flowing solid or in the form of a viscous product. The residual solvent content is typically reduced to a level of about 0.1 % in this process. The process flow scheme illustrated in Figure 15 provides for almost complete recovery of a solvent in on step, and thereby minimizes both disposal costs and nw solvent purchase costs. Systems are capable of handling very difficult materials ranging from highly viscous ands sticky products to crust-forming solid-like materials. Other typical examples are in the reprocessing of off-spec polymer and in processing paint sludge
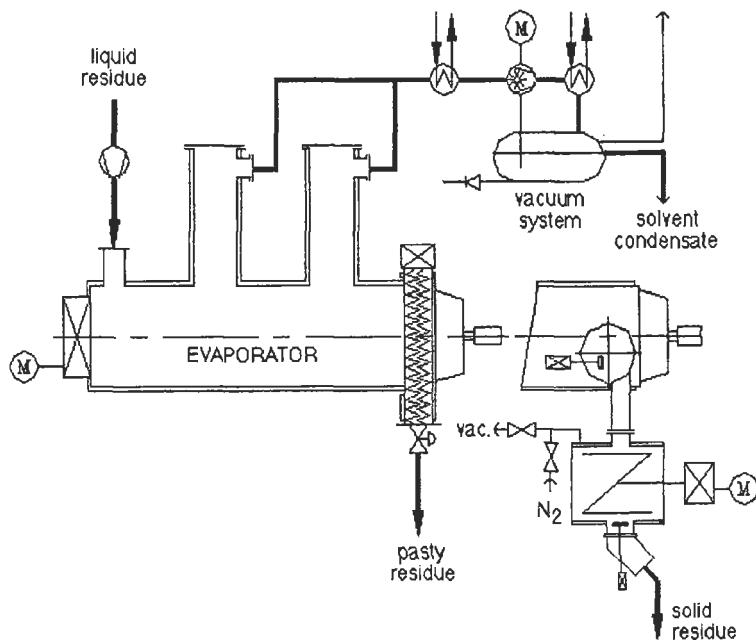
Figure 15. *Process flow scheme for DMAC recovery by evaporation.*
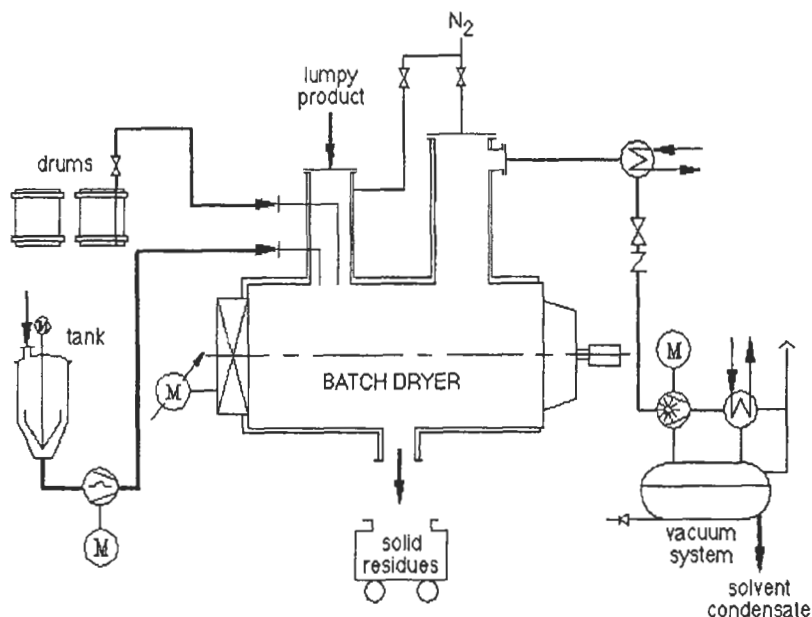
Figure 16. *Process flow scheme for a batch vacuum drying operation in the processing of rubber wastes.*

An example of continuous evaporation is in the processing of synthetic rubber solutions containing EPDM, SBr, Br, and the like. In the continuous evaporation of synthetic rubbers, a metered solution is passed through a preheater. The heated solution is then flashed into an evaporator where the remaining solvent is evaporated upon contact by heat. The viscous rubber is then discharged through a twin screw extruder, which serves for final stage drying of the rubber product. The example, shown in Figure 16, can be describes as direct, single-stage devolatization. This process has an advantage over more conventional rubber finishing (i.e., drying and solvent recovery) operations. More conventional systems use a combination of steam stripping, expeller/expanding/drying equipment and steps, as opposed to the single-step desolventizing process illustrated in Figure 16. As such, there are both energy savings, as well as environmental credits with a more compact, closed system. Typical applications include the processing of atactic polypropylene, elastomers, adhesives, silicon mass, and ABS/ATS polymers.

Another example in the polymers industry is illustrated in Figure 17, which is a process aimed at the batch drying of waste residue with solvent recovery. In this application liquid or viscous waste solutions are pumped into a batch dryer where they are dried under vacuum to a solid granular residue. Vaporized water and solvent are recovered by condensation and then separated by gravity. The process scheme is flexible, offering a range of temperatures and vacuum levels for treating

very sticky and crusty residues. It provides nearly complete solvent recovery and yields inert solid residues, thereby minimizing air pollution emissions. Chemical conditioning of the residue also occurs in the process. Typical applications can include the preparation of washing solutions, processing of paint residues, process residues, oil sludges, and contaminated oil sludges.

An example of the use of evaporating equipment in the food processing industry is illustrated in Figure18. This is an example of continuous vacuum drying to produce milk crumb. Preconcentrated liquid material is continuously crystallized and dried through a viscous, pasty intermediate phase until a crumbly product is discharged via a piston lock system. Maillard-type reactions impart special flavoring to the final product. Exact temperature control produces a high-quality, good tasting product. The compact design for this system features a clean-in-place system that requires little floor space. The process provides safe processing during sticky, crust-forming, intermediate stages of product processing.

The system shown is heavily used in food processing applications such as milk or chocolate crumb production, sugar substitutes, modified starch, and alginates. In addition to food processing applications, such a system is used in the processing of heat-sensitive pharmaceuticals, polymer suspensions like latex, in processing pigments and dyestuffs, and pesticides.
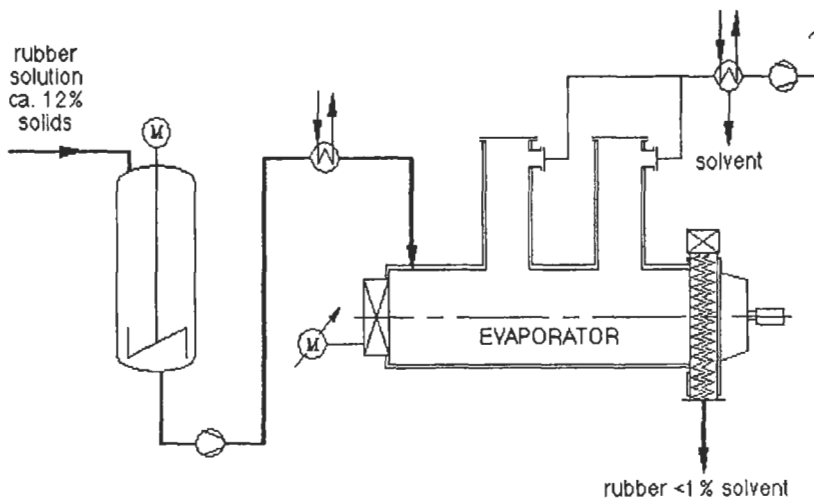
Figure 17. *Process flow scheme for continuous evaporation in a rubber desolventizing process.*
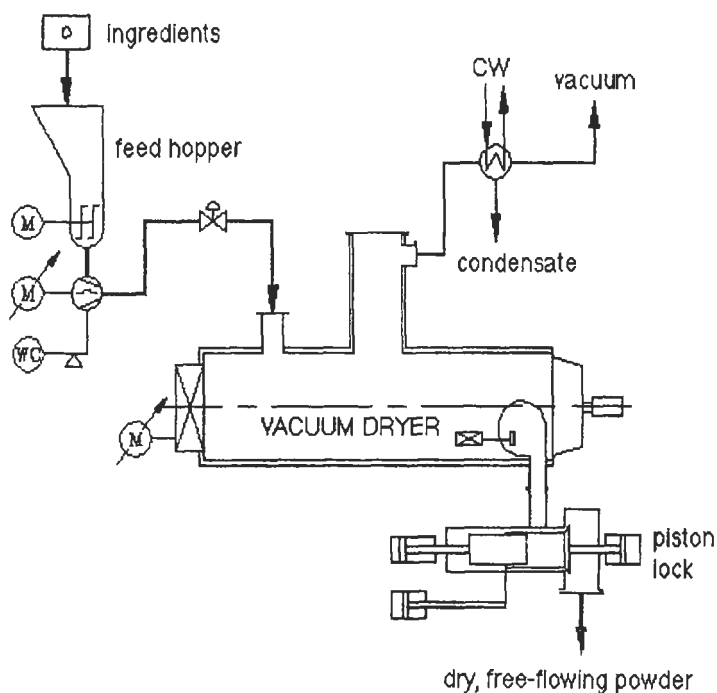
Figure 18. *Process flow scheme for continuous vacuum drying in the food processing industry.*

An example of continuous contact drying of residues and sludge is shown in Figure 19. This process is designed for handling paint sludge. The sludge is homogenized and metered to the continuous dryer by a paste hopper-feed screw system, or by special piston pumps. The residue is continuously dried through a viscous paste phase in a single step without recycling.

The resulting dried residue is continuously discharged through a twin screw or rotary valve. Solvent and vapors are condensed and recovered. The system shown is versatile and can process washing and cleaning solutions, sticky residues from chemical, pharmaceutical and food processing applications, contaminated soils, and oil sludge, in addition to paint sludge.

An example in the pharmaceutical industry is shown in Figure 20, which is a batch contact drying application. In drying pharmaceuticals. The filter cake is batch dried at low temperatures under vacuum to a free flowing product. Solvent vapors leave through a dust filter. The solvent condensate passes through an indirectly-cooled, solvent ring-pump, and is recovered. Additional applications include the processing of pigments and dyestuffs, temperature sensitive pharmaceuticals and foods, polymers and additives, various residues, sludge and salts.
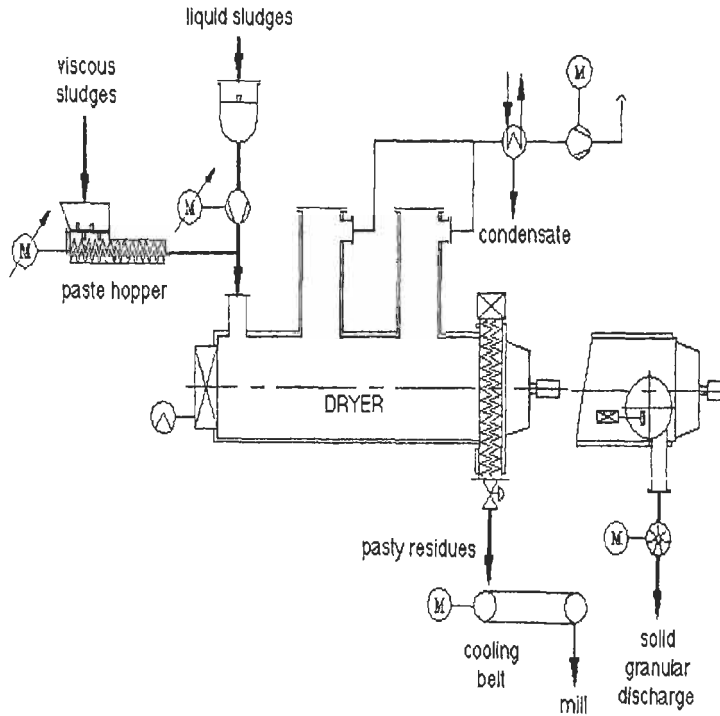
Figure 19. *Process flow scheme for continuous contact drying of residues and sludges.*
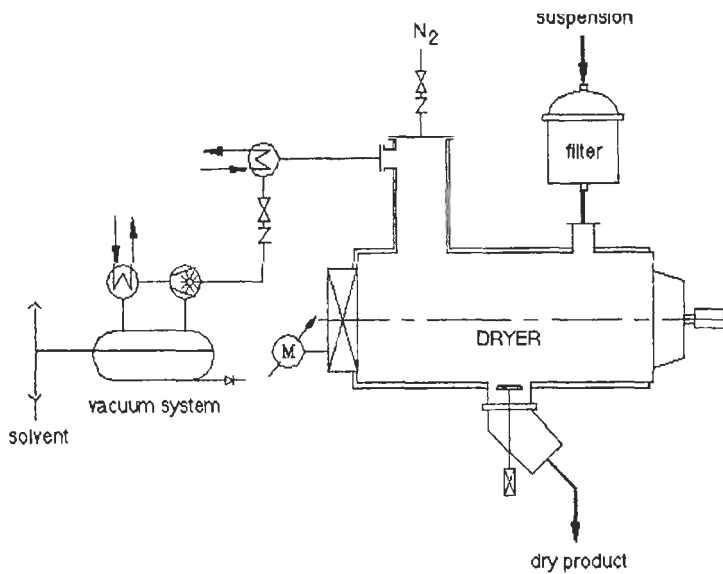
Figure 20. *Process flow scheme for batch contact drying application.*

Energy Requirements and Multiple Effects

The change in free energy that is required in evaporation is a measure of the theoretical difficulty of carrying out any reaction or separation, which is the minimum amount of work necessary to effect the change involved, if performed under theoretically perfect and reversible conditions. Any process should be able to operate with a work consumption that does not greatly exceed this value. It is important to recognize that this energy must be supplied as work. In other words, heat in itself is not the equivalent of energy, although for convenience energy is often expressed in heat units. From thermodynamic considerations it can be shown that the minimum amount of work required is very small, amounting, in the case of a 10 per cent water solution of glycerin, to a mere 6.4 Btu per lb of water evaporated. Obviously the more concentrated the solution, the greater is the theoretical energy required, but even for concentrated solutions this value remains relatively small. For colloidal solutions, i.e., those of high molecular weight, the reversible work is even less. Theoretically, therefore, the greater part of the heat supplied in evaporation should be recoverable, and such is the case in real life.

Since the free energy necessary is work and not a mere heat requirement, available heat engines actually convert 25 per cent or more of the heat supplied into work. Hence, the theoretical heat requirement for a 10 per cent glycerin solution is only 32 Btu per lb of water evaporated. From this point of view, a simple evaporator is a very inefficient system. However, the heat required for evaporation is almost entirely converted into latent heat of vaporization, and is, in this form, carried out of the evaporator by the vapor and can be again utilized by condensing the vapor. A simple example of this is a plant operation where a certain amount of water is being evaporated at atmospheric pressure in the process of concentration of an aqueous solution. Most of the heat consumed appears as latent heat in the water vapor. It could not, however, readily and directly be employed to evaporate more water under these conditions, since on condensing it this heat is evolved at the boiling point (100°C.). As such, no temperature difference exists to force the heat through the necessary heating surface into the fresh solution to be evaporated. If, however, the same plant operation had a corresponding supply of alcohol to be distilled, the water vapor from the evaporator could readily be used as a heat supply therefore, by condensation in the heating coils of the alcohol still, rather than by using boiler steam for the still.

If, in turn, this same plant had ether to be distilled, the heating coils of the ether still could be used as a condenser for the alcohol vapor, thus avoiding the use of boiler steam in the ether still. In this way a succession of liquids of progressively lower boiling points can be evaporated with a single supply of heat. In other words, in the process of evaporation, the heat used is merely degraded in temperature and this degradation is for all practical purposes slight.

This leads us to a discussion on multiple effects. The above conditions enable a given quantity of heat to be used for evaporation a multiple of times by boiling a series of liquids of successively lower boiling points. This is a very practical application throughout the CPI. It is important to note that the boiling point of a water solution depends upon the pressure above it, and by a careful selection of pressures a solution can be made to boil at the temperatures given for alcohol and ether or any more suitable temperatures, and a similar economy of heat can be effected. Each evaporator is referred to as an effect, and the overall process is called multiple-effect evaporation. We consider the classic example given by Walker, Lewis, McAdams and Gilliland (1937), shown in Figure 21, whereby the steam entering the coils of effect A is saturated exhaust steam at 2.5 lb. gauge (17.2 lb. per sq. in. abs. pressure) and at 220 °F, and the pressure in the vapor space of A corresponds to a vacuum of 8.75 in. of mercury, or with normal barometer 10.4 lb per sq.in.(abs) pressure. The water in A will boil at 195 °F and furnish steam to the heating coils of B at this temperature. The vacuum in the vapor space of B is 19.1 in. and hence water will boil at 165 °F. The vapor coming from B and entering the heating coils of C at 165 °F will cause water to boil in C at 125 °F where the vacuum is 26 in. Vapor from C at 125 °F will then pass to the condenser. Since the heating element of A is above atmospheric pressure, the condensed water in it can flow from it by gravity at a little below 220 °F. But the condensed water in the heating space of B is under vacuum; hence the pump P' must be used to exhaust the heating space in B and deliver the water at atmospheric pressure. Similarly, the condensate in the heating space of C must be pumped out. The condensed water from the heater in B at about 195 °F is in position to give up part of its sensible heat to the liquid boiling in C at 125 °F, and by leading it into the heating element of C part of it flashes into steam due to the lower pressure, thereby reducing its temperature to 165 °F (the temperature of the steam in the coils of C).
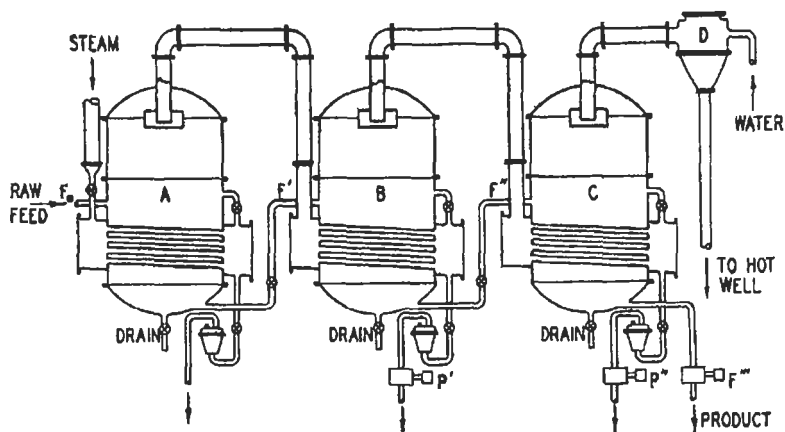
Figure 21. *Multiple effect evaporator.*

The hot condensate from the heater in A can be returned to the boiler. The solution to be evaporated is fed at 195 °F into the liquor spaces of effect A at F_o by means of a pump or by gravity. Since the pressures in B and C are progressively lower, the solution can be drawn in sequence through the system by the pipes F' and F" and finally lifted to atmospheric pressure by the pump F'''. This arrangement is called parallel flow, or forward flow. In order to permit the removal of fixed gases from the heating space of all the effects, each is connected by a small pipe to the vacuum condenser or to the body of the vapor space of the same effect which is obviously at lower pressure. Where the noncondensable gas is small, the latter is better practice, as this, method acts as a safeguard against the loss of steam due to its discharge with fixed gases.

In the above example, 1 lb of initial steam should evaporate approximately 1 lb of water in each of the effects A, B and C. In practice however, the evaporation per pound of initial steam, even for a fixed number of effects operated in series, varies widely with conditions, and is best predicted by means of a heat balance. This brings us to the term *heat economy*. The heat economy of such a system must not be confused with the evaporative capacity of one of the effects. If operated with steam at 220 °F in the heating space and 26 in. vacuum in its vapor space, effect A will evaporate as much water (nearly) as all three effects costing nearly three times its much; but it will require approximately three times as much steam and cooling water. The capacity of one or more effects in series is directly proportional to the difference between the condensing temperature of the steam supplied, and the temperature of the boiling solution in the last effect, but also to the overall coefficient of heat transfer from steam to solution. If these factors remain constant, the capacity of one effect is the same as a combination of three effects.

It is important to note that the highest temperature employed in an effect is limited by the corresponding pressure, which must not exceed the working strength of the apparatus. At times it is, of course, limited by the pressure of the available steam supply, and also by the temperature to which the solution may be safely heated (i.e., without destroying its properties). The lowest pressure that can be realized depends upon the temperature of the cooling water. These limiting temperatures, together with the nature of the solution to be evaporated and the product required, are factors not usually within the control of the designer of evaporative equipment, but are already determined. Collectively, they are designated the "terminal conditions" of an evaporative system, and represent the fixed and accepted limitations under which the equipment must be designed to operate.

Finally, we may note that every solution exerts a vapor pressure less than that of the pure solvent at the same temperature. Corresponding to this vapor-pressure lowering is an equivalent boiling-point raising. Dissolved substances lower the vapor pressure of the solvent. Such reduction increases with the concentration of solute. Since a solution boils when its vapor pressure reaches that of its surroundings, it must be heated to a temperature above the boiling point of the pure

solvent before ebullition can take place. The vapor rising from a solution is therefore superheated by an amount equal to this boiling-point raising (b.p.r.) and it must be cooled by this amount before it will condense and give up its heat of vaporization. While the amount of superheat is small when compared to the latent heat of the vapor, it should be noted that this latent heat of vaporization becomes available for use only when the vapor is cooled to a temperature lower than that of the solution from which it came by the amount of this b.p.r. This loss of temperature difference from condensing steam to boiling liquid in the next effect increases the heating surface required for the transfer of the heat.

Flow Regimes

In gas-liquid flow the two phases can be distributed in the equipment in many configurations called flow regimes or flow patterns, differing from each other in the spatial distribution of the interface. The flow pattern depends on the operational variables, physical properties of the fluids, and geometrical variables of the system. Hydrodynamics of the flow, as well as the mechanisms of momentum, heat and mass transfer, change significantly from one flow regime to another. As a result, accurate understanding of any process in two-phase flow depends on the knowledge of the existing flow patterns, and determining the flow pattern is the first step for developing techniques to accurately predict pressure drop, heat and mass transfer, gas and liquid holdup, etc. - the basic parameters needed in sizing evaporators. The flows observed may take many different configurations with respect to the distribution of the gas-liquid interface. Transitions between the various configurations are usually gradual with respect to the flow rate changes or the pipe inclination. The main task in classifying the flow patterns is to group together flow configurations that have common character according to some defined definitions. The designation of the flow pattern should be based on the flow configuration that has basically the same character, pertaining to the distribution of the interfaces and the mechanisms dominating pressure drop and heat and mass transfer. We will consider only the most standard flow regimes encountered in evaporative systems, and examine them only from the standpoint of simply geometry flows, such as in pipes. Typical flow regimes encountered in horizontal pipe flow configurations are summarized in Figure 22. A brief description of these follows:

Stratified (S) - Liquid flows at the bottom of the pipe with gas at the top. The stratified pattern is subdivided into stratified smooth (SS) where the liquid surface is smooth, and stratified wavy (SW) where the interface is wavy.

Intermittent (I) - In this flow pattern the liquid inventory in the pipe is non-uniformly distributed axially. Plugs or slugs of liquid that fill the pipe are separated by gas zones that contain a stratified liquid layer flowing along the bottom of the

pipe. The liquid may be aerated by small bubbles, which are concentrated at the front of the liquid slug and the top of the pipe. The intermittent pattern is usually subdivided into slug and elongated bubble flow patterns. The elongated bubble pattern is considered the limiting case of slug flow when the liquid slug is free of entrained gas bubbles. With this criterion, the elongated bubble-slug transition is quite sharp and easy to identify.

Annular (A) - The liquid flows as a film around the pipe wall. A liquid film surrounds a core of high velocity gas that may contain entrained liquid droplets. The film at the bottom is normally thicker than at the top depending upon the flow rates of the liquid and gas phases. At low gas rates at which a transition to annular flow from slug flow is generally observed, most of the liquid flows at the bottom of the pipe. The upper walls are intermittently wetted by large aerated, unstable waves that sweep through the pipe. This is not slug flow, which requires a competent bridge of liquid, nor is it considered to be fully developed annular flow, which requires a stable film over the entire pipe perimeter. It is therefore designated as the wavy annular pattern, or sometimes called "proto-slug" flow.
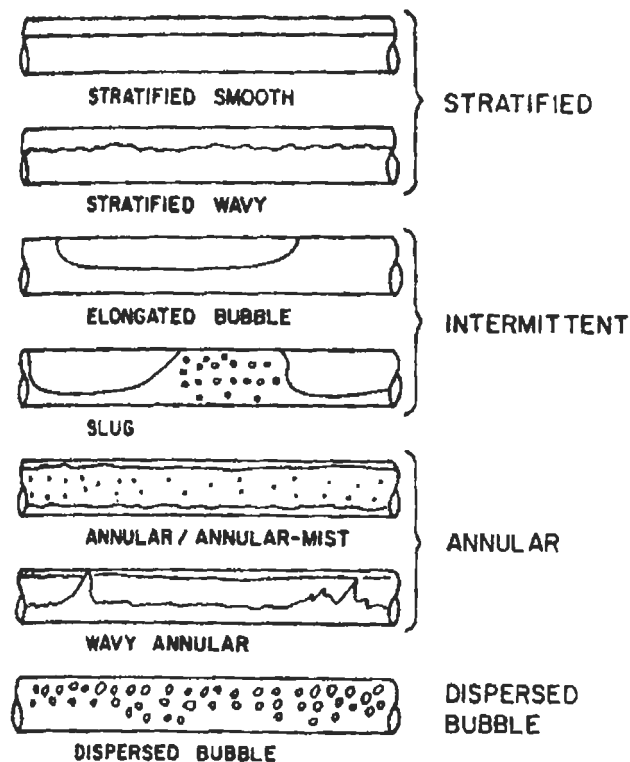
Figure 22. *Flow patterns in horizontal flow.*

Dispersed bubble (DB) - The gas phase is distributed as discrete bubbles within a continuous liquid phase. At the transition boundary most bubbles are located near the top. As the liquid rate increases, the bubbles are more uniformly dispersed.

The flow patterns typically encountered in vertical pipe flow are illustrated in Figure 23. The types of flow patterns encountered are as follows:

Bubble flow - The gas is roughly uniformly distributed in the form of small discrete bubbles in a continuous liquid phase. The flow pattern is designated as bubble flow (B) at low liquid flowrates, and as dispersed bubble (DB) at high liquid flow rates in which case the bubbles are finely dispersed in the liquid.

Slug flow (SL) - Most of the gas is located in large bullet-shaped bubbles that have a diameter close to that of the pipe diameter, and move upward. The bubbles are separated by slugs of continuous liquid that bridges the pipe and usually contains small gas bubbles. Between the bubbles and the pipe wall, liquid falls downwards in the form of a thin falling film. When the flow is low there appears well-defined gas-liquid boundaries, and the liquid slug is free of bubbles. This case is often referred to as plug flow. When the flow is faster, boundaries are not very clearly discerned and froth is generated, and the term slug flow is used.
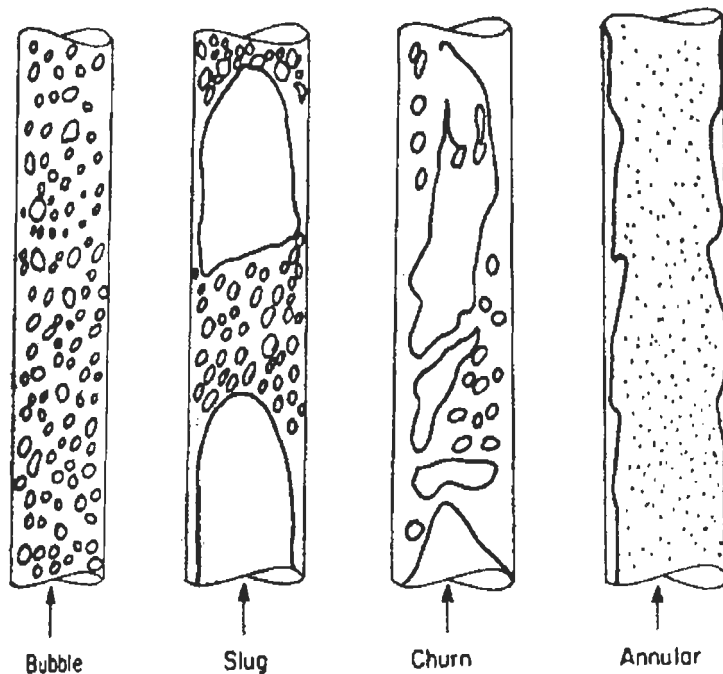
Figure 23. *Flow patterns in vertical pipe flow.*

Churn flow (CH) - This is similar to slug flow, but is much more chaotic, frothy, and disordered. The bullet-shaped bubble becomes narrow and its shape is distorted. The continuity of the liquid in the slug between successive bubbles is destroyed by a high gas concentration in the slug. As this happens, the liquid the liquid slug falls. This liquid accumulates, forming a bridge, and is again lifted by the gas. Characteristic of churn flow is an oscillatory motion of the liquid slug. For higher rates the flow is even more disordered and is sometimes called froth flow. Annular flow (A) - The flow is characterized by the continuity of the gas phase along the pipe in the core. Liquid phase moves upwards partially in the form of drops entrained in the gas core. The annular flow is termed wispy annular when the entrained phase is in the form of large lumps or "wisps". The terms froth, mist, or semi-annular describe conditions between churn and annular.

In vertical downward flow as well as in upward and downward inclined flows, the flow patterns that can be observed are essentially similar to those described above, and the definitions used can be applied. Experimental data on flow patterns and the transition boundaries are usually mapped on a two dimensional plot. Two basic types of coordinates are generally used for this mapping - one that uses dimensional coordinates such as superficial velocities, mass superficial velocities, or momentum flux; and another that uses dimensionless coordinates in which some kind of dimensionless groups are used as coordinates. The dimensional coordinates maps are inherently limited to the range of data and flow conditions under which the experiments were conducted. In spite of this limitation, it is widely used because of its simplicity and ease of use. Figure 24 provides an example of such a map.
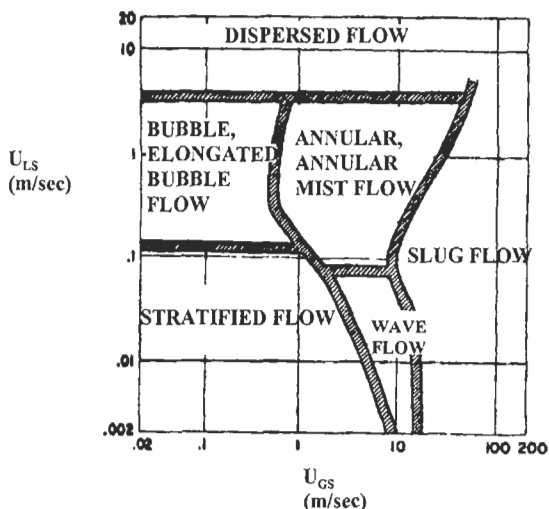
Figure 24. *Example of flow pattern map for air water system in horizontal pipes.*

Maps based on dimensionless coordinates using coordinates such as the Froude number, gas volumetric ratio, Reynolds number, liquid, and gas mass flow rate ratio have been suggested by numerous investigators. Figure 25 shows a map that uses as coordinates $(V_T/\sqrt{g}D)^{1/2}$ versus Q_L/Q_G, where Q_L and Q_G are the liquid and gas volumetric flow rates, respectively. V_T is the average total velocity in the pipe, D is the pipe diameter, and g the acceleration of gravity. Other systems employ a mixed coordinate system. For example, the well known Baker plot (Figure 26) uses G/λ versus $L\lambda\psi/G$, where G is the gas mass velocity (a dimensional coordinate) and L/G is a ratio of liquid and gas mass flow rates. The fluid properties enter as the dimensionless correction factors defined by the following expressions:

$$\lambda = [(\rho_G \, / \, 0.075) \, (\rho_L \, / \, 62.3)]^{1/2}$$

$$\psi = (73.0/\sigma) \, [(\mu_L \, / \, 1.0) \, (62.3 \, / \, \rho_L)^2 \,]^{1/3}$$

where ρ_G and ρ_L are the gas and liquid densities (in units of lb/ft^3), μ_L is the liquid viscosity (centipoise), and σ is the surface tension (dynes/cm). Flow pattern maps that are based upon dimensionless coordinates are generally thought to be more reliable since they are based upon more universal parameters and are independent of fluid properties and system geometric parameters. However, dimensionless plots are not always easy to construct and the selection of the proper parameters for dimensionless groups is not trivial.
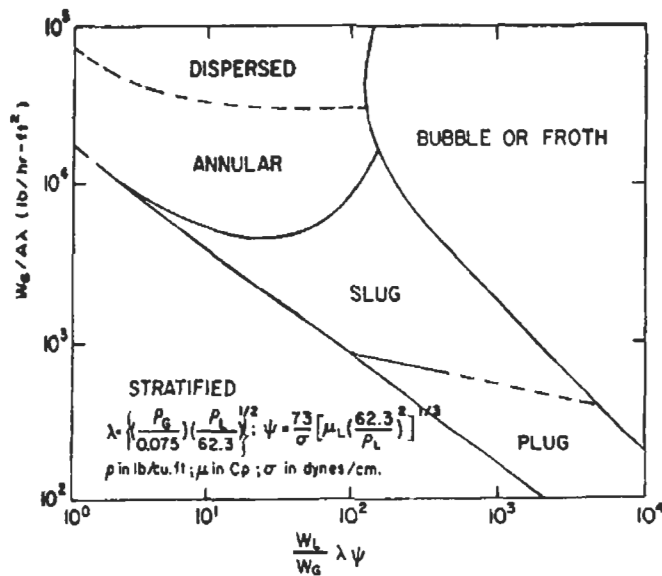
Figure 25. *Baker flow map.*
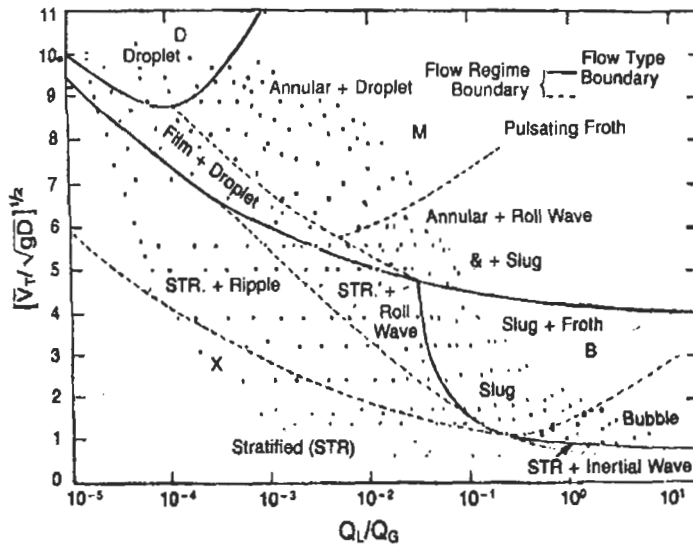
Figure 26. *Flow regime map for horizontal gas and liquid flows (D - droplet; X - stratification; M - mixed; B - bubble).*

Dimensional analysis is a straightforward approach to select the proper dimensionless coordinates. The approach basically involves guessing what the physical variables that affect the flow patterns are. Under isothermal incompressible conditions these are the liquid and gas flow rates (or the superficial velocities U_{Ls} and U_{Gs}), the liquid and gas densities and viscosities (ρ_L, ρ_G, μ_L and μ_G), pipe diameter (D), pipe roughness (ϵ), pipe inclination angle (β), acceleration of gravity (g), liquid surface tension (σ), and the solid to liquid gas contact wetting angle (γ). The flow pattern transition boundaries should be a function of all these variables. Note that there is already a reduced set of variables as it is assumed that the entrance geometry has no influence on the steady state flow pattern. Using the Buckingham π method one can show that the flow pattern boundaries depend on the following dimensionless parameters:

$$(U_{Ls}\rho_L D)/\mu_L, \ (U_{Gs}\rho_L D)\mu_L, \ g\rho_L^2 D^3/\mu_L^2, \ \rho_G/\rho_L, \ \mu_G/\mu_L, \ \sigma \rho_L D/\mu_L^2, \ \beta, \ \epsilon/D, \ \gamma$$

Note that this list of dimensionless parameters is by no means unique. A set of variables in which each variable in the new set is a combination of the abovementioned set is also permissible and is in principle completely equivalent to the original set. In fact, an infinite number of sets of the dimensionless parameters exist, each of which could be justified as the "original" set. The normal approach at this point is to find an explicit functional relation among one set of variables

based on experimental data. To find the relation among nine parameters based only upon experimental data is impractical. Furthermore, the rigorous result is not a two-dimensional map but rather a nine-dimensional map. In order to reduce the number of dimensionless groups one tries to distinguish among the important and non-important variables. The correlation of the data is performed with relation to the important variables in the hope that the number of important parameters will be only two or three so that the result could be plotted on a two-dimensional map. Note that the number of important parameters depends also on the choice of the dimensionless set of variables. An alternative set of parameters, for example, is:

$$(U_{Ls} + U_{Gs})/(gD)^{1/4}, \ U_{Ls}/U_{Gs}, \ g\rho_L^2 D^3/\mu_L^2, \ \rho_G/\rho_L, \ \mu_G/\mu_L, \ \sigma\rho_L D/\mu_L^2, \ \beta, \ \epsilon/D, \ \gamma$$

In addition to flow regime, hold-up and pressure drop are two other important parameters in two-phase gas-liquid flows. Hold-up is defined as the relative portion of space occupied by a phase in the pipe. It can be expressed on a time or space average basis, with the actual method chosen depending on the intended use of the hold-up value, and the measurement method employed. There are numerous correlations in the literature for hold-up, but most are based upon a pressure drop-hold-up correlation. The following expression is a widely recognized empirical relationship between hold-up and pressure drop:

$$(dP/dL)_F/(dP/dL)_{SL} = a/(1-R_G)^b = \phi_L^2$$

A plot of $(dP/dL)_F/(dP/dL)_{SL}$ versus $(1-R_G)$ results in three distinct lines shown in Figure 27, where the values of the constants a and b are indicated. The three ranges correspond to approximately the bubble-slug, stratified, and annular flow regimes. Formulas to calculate R_G for substitution into the above equation are:

$$(1-R_G) = [4+(8/7)d_1]/[5+d_1(\beta/(1-\beta))+8/7]$$

where

$$d_1 = 4[0.69 +(1-\beta)(4+21.9(V_{LS}/(gD)^{1/2}]Re_{SL}^{1/8} (\rho_G/\rho_L)^{1/2}$$

An extensive treatment of gas-liquid flows encountered in industry applications, along with numerous design correlations can be found in Volume 3 of the Encyclopedia of Fluid Mechanics - Gas-Liquid Flows (N. P. Cheremisinoff, editor, Gulf Publishing Co, Houston, TX, 1986). Further discussions in this volume can be found in Chapter 4 with regard to flow regimes typically encountered in bubble columns and similar devices.
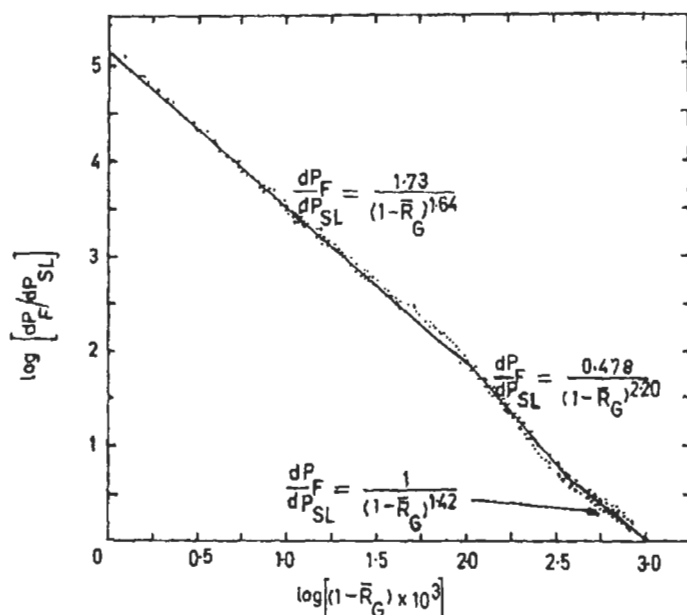
Figure 27. *Empirical correlation between pressure drop and hold-up.*

DRYING EQUIPMENT

Drying Mechanisms

Drying is a basic unit operation that is applied as a pretreatment step, an intermediate step, or as a finishing step in the preparation of different products. Although drying operations sometimes have a specific objective in a few specialized industries, in general applications throughout the CPI the objective is the removal of water from a system or structure, when the amount of water present is comparatively small. In the immediate discussions we focus only on the most common applications, whereby the material to be dried may be either a gas, a liquid or a solid. If we are handling a gas, the water may be carried simply as vapor, or in addition it may be present in the form of a spray, fog or mist. If a liquid, water is removed by drying only when present in small amounts; otherwise the process is described as evaporation or distillation. For example - alcohol or acetone may be "dried" by removing the water therefrom. In contrast, for solids the amount of water may vary from a mere trace to the large amount present as in fresh fruit or

in a glue jelly. As the moisture leaves a wet solid there is a tendency for the structure to undergo volume reduction, or to shrink. Hence, if the process is not carefully controlled, shrinkage will not be uniform, and cracking or warping can result, thereby deteriorating the product. Examples of this action are found in timber and unburned pottery. Still another example is if a material is dried under tension and lateral shrinkage is prevented, the structure loses its strength. Such is the case when paper is dried on certain machines as compared to that slowly dried in a loft. Many materials such as eggs, fruit or milk must be dried rapidly at as low a temperature as is practical, in order to preserve their characteristic flavor and the other valuable product properties they possess. Some substances deteriorate, when allowed to remain for a considerable length of time in a wet condition. Material such as glue, starch and sugar cannot be heated while wet owing to the solvent action of the water upon them.

There are many materials containing water of crystallization that simply cannot be dried under ordinary conditions. These materials would loose all or a portion of this water. Under such widely varying conditions drying must be accomplished in a drier that takes into consideration the rate of drying, the condition of the drying medium, and the nature of the exposed surfaces of the material. Each of these factors is complex and requires careful engineering.

The standard methods of drying can be classified as deposition of the moisture as either water or ice; decomposition of the water; chemical precipitation; absorption; adsorption; mechanical separation; and vaporization. The completeness with which dryness can be accomplished by any process depends upon the factors controlling the equilibrium conditions achieved in the operation. A brief discussion of each method is first given.

For deposition of the moisture as water or ice - when condensation takes place in a gas, it is the reverse of vaporization and depends upon the same fundamental considerations. This is often described by the term dehumidification. Water may be removed from liquids by converting it into ice and in this form separating it from the portion having a lower freezing point. The alcoholic content of a liquid possessing a delicate flavor can, in this way be readily increased. Fruit juices are concentrated by cooling with agitation and subsequently separating the ice crystals by passing through a centrifugal machine. The amount of moisture remaining in air dried by cooling depends upon the temperature to which it is cooled. Also in the case of a liquid it depends upon the temperature reached, but is limited when a eutectic mixture is formed.

In the case of decomposition of the water - when a small amount of water is found in either a gas or a liquid, it may be removed by decomposing it and separating the products formed. For example, ether can be dried by placing it in contact with metallic sodium. In the reaction, caustic soda is formed, which clings to the surface

of the metal and, if separated, will eventually settle out of solution. Separation can be enhanced by the addition of carbides and nitrides, however, the completeness of the process is largely a function of mechanical agitation of the liquid and the reacting substance. The vapor pressure of water with which the substances formed are in equilibrium is so small as to have negligible impact.

Chemical Precipitation - In this case, larger amounts of water may be advantageously withdrawn in the form of a chemically combined precipitate. Lime forms calcium hydroxide; anhydrous sulfate of copper, or sodium takes oil water of crystallization and separates from the liquid. The amount of water remaining in the liquid is controlled by the vapor pressure of the hydroxide or the crystalline substance formed. Phosphorus pentoxide and calcium chloride may be used for drying gases, and for drying liquids in which they are insoluble; again the completeness of drying is limited by the vapor pressure of the resulting hydrated substance.

Absorption - Processes water can be removed from a material by the capillary action of porous bodies. An example is the cream of clay and water used for casting pottery, which is deprived of the greater part of its water by placing it in molds of plaster of Paris. The capillary character of this mold withdraws the water from the liquid clay mixture and deposits upon itself a layer of solid clay, the thickness of which is controlled by the time of standing. Certain types of candies, such as gumdrops, are dried mainly by contact with the starch molds in which they are cast. The drying effect of sponges, towels and materials of this kind is due to this same action.

Adsorption - The ability of certain substances to adsorb moisture makes possible the application of this process to drying gases, and to some extent also liquids. The process of adsorption is described in greater detail later on.

Mechanical Separation - Materials which are of a spongy nature and capable of holding by capillary action large quantities of moisture, can be readily expelled by pressure alone. When such is the case, it is evidently desirable to get rid of as large a percentage of moisture as possible by such means before passing to other more expensive methods. Examples where this may be accomplished include the drying of various textiles, wool, grains, whereby centrifugation is often used for the moisture separation.

Vaporization - The process for drying liquids and solids generally involves vaporizing the water as a first step; and then separating the water from the structure of which it initially formed a part of. If air or some inert gas is used to carry away the water vapor formed, the process is simply referred to as air drying. If the vapor is passed through a condenser, and a pump is used to withdraw the air from the system, then the operation is referred to as being vacuum drying. Essentially all solids and liquids exert at any definite temperature a perfectly definite vapor pressure. If the material we are working with is a mixture, each component has its

own specific vapor pressure. These individual or partial pressures are additive and collectively make up the vapor pressure of the substance. If the component parts of a mixture are mutually soluble, the partial pressure of each component will be thereby somewhat decreased. The vapor pressure of liquids increases as the temperature rises and will be some definite temperature at which this vapor pressure equals atmospheric pressure. The vapor of the liquid will continue to form at this stage, and not only diffuse but push back the atmosphere into the surrounding space, maintaining as total pressure the atmospheric pressure then existing. The liquid at this point boils and this temperature the material's boiling point. As the pressure of the atmosphere varies, the boiling point of a liquid will vary also. These fundamental principles are essential to the process of vaporization. This leads us to some fundamental terms that are the foundation of drying technology.

The term *humidity* refers to the amount of water vapor present in a gas. The design of industrial equipment such as air conditioning systems, the drying of solids by gases, the drying of gases by liquids, the cooling of liquids by their evaporation into gases (e.g., cooling towers, spray ponds, etc.) depends upon a clear conception of this quantity. The conventional system of units used to express humidity is based on one part by weight of dry air. Of course any unit may be employed, however we will use the pound for illustrative purposes. Humidity (H) is simply defined as the number of pounds of water vapor carried by 1 lb of dry air; and the term absolute humidity is often applied. The per cent absolute humidity is the number of pounds of water vapor carried by 1 lb of dry air at a definite temperature, divided by the number of pounds of vapor that 1 lb of dry air would carry if it were saturated at the same temperature. In other words, it is the humidity of air at any temperature expressed as per cent of the humidity of saturated air at the same temperature.

The percent relative humidity is defined as $100p/P_s$, where p is the actual partial pressure of the water vapor and P is the vapor pressure of water at the same temperature. The total pressure is taken as normal barometric, unless otherwise stated. Note that since the per cent relative humidity is defined as $100(p/P_s)$ and the per cent absolute humidity equals $100[p/(760-p) \div P_s/(760-P_s)]$, the factor by which the former must be multiplied to convert it to the latter is $(760 - P_s) / (760-p)$, where p and P_s can be expressed in units of millimeters of mercury.

The term *dew point* (also known as the saturation temperature, t_s) is the temperature at which a given mixture of air and water vapor is saturated with water vapor. More simply stated, it is the temperature at which water exerts a vapor pressure equal to the partial pressure of the water in the air.

The term *humid heat* (s) is the number of Btu necessary to raise the temperature of 1 lb of dry air plus such water vapor as it contains by 1 °F. Humid heat obviously increases with increasing vapor content of the air, and is therefore a function of

humidity. Since over the range of conditions involved the specific heats of dry air and water vapor are substantially, constant, being 0.24 and 0.45, respectively, the following expression can be used to estimate the humid heat:

$$s = 0.24 + 0.45H$$

where H is humidity.

Humid volume is the volume in cubic ft of 1 lb of dry air together with the water vapor it contains. It is influenced by temperature, pressure and humidity.

Saturated volume is the volume in cubic feet of 1 lb of dry air when it is saturated with water vapor; that is, it is the humid volume at saturation, and is determined by the temperature and pressure. The humid volume of air equals the product of its saturated volume at its dew point and the ratio of the absolute temperature of the air to the absolute temperature of its dew point.

Figure 28 shows the key features of the humidity chart. The chart consists of the following four parameters plotted as ordinates against temperature on the abscissas: (1) Humidity H, as pounds of water per pound of dry air, for air of various relative humidities; (2) Specific volume, as cubic feet of dry air per pound of dry air; (3) Saturated volume in units of cubic feet of saturated mixture per pound of dry air; and (4) latent heat of vaporization (r) in units of Btu per pound of water vaporized. The chart also shows plotted humid heat (s) as abscissa versus the humidity (H) as ordinates, and adiabatic humidification curves (i.e., humidity versus temperature). Figure 28 represents mixtures of dry air and water vapor, whereby the total pressure of the mixture is taken as normal barometric. Defining the actual pressure of the water vapor in the mixture as p (in units of mm of mercury), the pressure of the dry air is simply 760 - p. The molal ratio of water vapor to air is p/(760-p), and hence the mass ratio is:

$$H = p(18.02)/[(760-p)(28.97)]$$

whereby H is expressed in units of pounds of water vapor per pound of dry air.

Specific volume is determined by application of the ideal gas law. One pound mole of air occupies a volume of 359 cubic feet at standard conditions, hence:

Specific volume = $(359)(t+460)/\{(28.97)(492)\} = 11.57 + 0.0252t$

where temperature t is in units of °F.

As defined earlier, the saturated volume is on a basis of 1 lb of dry air, and equals the sum of the specific volume of the dry air plus the volume of the water vapor

associated with the particular saturated mixture. The weight of the water vapor in saturated air at 100 °F is seen from the 100 per cent humidity line to be 0.0428 lb. The corresponding volume is:

$$v = (0.0428)(359)(460 + 100)/(18.02)(460 + 32) = 0.97 \text{ ft}^3$$

The saturated volume at 100 °F is then 14.09 + 0.97 = 15.06. A curve of such values is shown in Figure 28. Since humid volume is defined as the volume of any mixture, expressed as cubic feet of mixture per pound of dry air, and since both the specific and saturated values are on the basis of 1 lb of dry air, the humid volume for any humidity may be obtained directly by interpolation between the curves for specific and saturated volumes.

The latent heat of vaporization in Figure 28 is expressed in units of Btu per pound of water evaporated. The value is taken from the steam water vapor at various temperatures (i.e., from the steam tables).

Humid heat is defined by a linear relationship, s = 0.24 + 0.45H. The value of s for air, having an absolute humidity of 0.0222, is 0.24 + (0.45) × (0.0222) = 0.250. In Figure 28 the scale for s is shown at the top of the plot. Note that parameter s is the only variable that is not plotted against temperature.
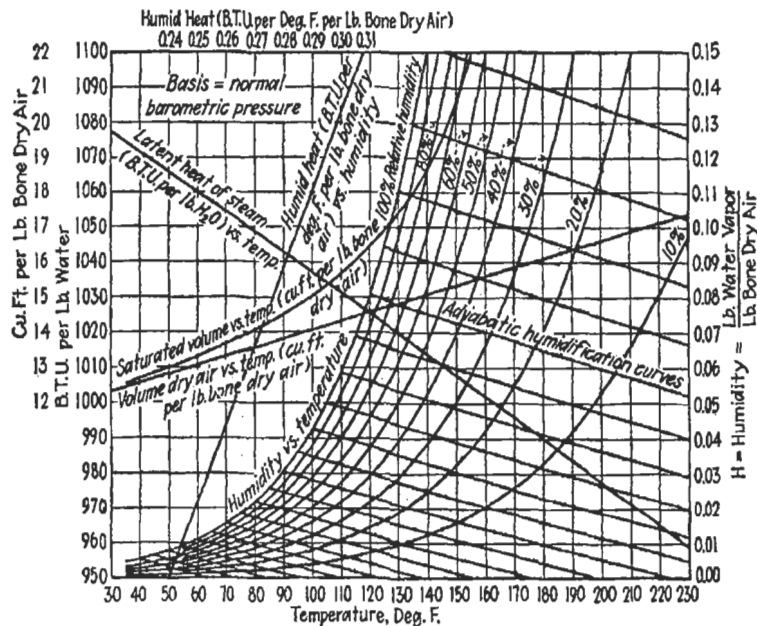
Figure 28. *Key features of the humidity chart.*

The elementary concepts discussed above are essential to a ready understanding of drying technology. Thus far, our discussion of the mechanism of vaporization of liquids suspended in air has shown the influence of the temperature, humidity and air velocity upon the rate of vaporization. When water is admixed with other materials, the composition and form of the mixture profoundly modify the evaporative rate. In order to define the effect of these variables, they will first be examined under constant drying conditions. Most solid materials that are to be dried exist in either sheet or lump form, with the water from which they are to be freed disseminated throughout the mass. The water (or any other liquid for that matter) may be physically held between the voids created by aggregates of individual particles, or physically held within a porous structure of individual particles, or in part, chemically bound to the surfaces of the solid particles. The mechanism of removal of this water is somewhat complicated, but must be thoroughly understood in order to appreciate the factors controlling the drying of such materials. We shall consider a simple system whereby focus is on the drying of a sheet material freely and equally exposed to the drying air on both sides of the sheet. The sheet will be assumed large in comparison with its thickness that the drying from the edge of the sheet may be neglected, as compared with the drying from the faces of the sheet. We may further assume that, initially, the water is uniformly distributed throughout the thickness of the sheet, and that it is only physically entrapped or coating the solid surface. As soon as the sheet comes into contact with the drying air, evaporation will start at the surface, and the concentration of water on the surface will therefore diminish. This will cause a difference in concentration between the interior of the sheet, which is still wet, and the surface, which has been partially dried by the evaporation process. Because of this concentration difference, there will be a flow of liquid water by diffusion from the interior of the sheet to the surface. These two processes, i.e., evaporation from the surface and diffusion through the sheet from the interior to the surface, will go on simultaneously until the drying operation is suspended or the sheet has come into equilibrium with the drying air.

In some cases, resistance to surface evaporation is the controlling factor in drying, while in others the resistance to diffusion of the water from the interior to the surface limits the rate of drying. In any case, it is essential to remember the part played by each of these processes in order to properly control a drying operation. The importance of these two processes is further explained by the following illustrations. Wet, green wood must be dried prior to use. During drying, the wood will shrink. If the surface evaporation is excessively rapid, the surface becomes very dry while the interior is still impregnated with moisture, i.e., a large gradient in moisture concentration from the interior toward the surface is created. The surface shrinks due to the evaporation of its moisture, and is subjected to high tension because of the incompressible character of the wet interior. If the surface shrinkage is excessive, this results in rupture of the surface with the generation of

cracks or checks which ruin the lumber for finer uses. In drying such lumber it is therefore essential to avoid creating too large a concentration gradient between the surface and the interior. This is accomplished by raising the humidity of the air to make the rate of surface evaporation commensurate with the rate of diffusion of water. Exactly the same applies to the drying of articles molded from wet clay in the ceramic industry.

Surface evaporation can be a limiting factor in the manufacture of many types of products. In the drying of paper, chrome leather, certain types of synthetic rubbers and similar materials, the sheets possess a finely fibrous structure which distributes the moisture through them by capillary action, thus securing very rapid diffusion of moisture from one point of the sheet to another. This means that it is almost impossible to remove moisture from the surface of the sheet without having it immediately replaced by capillary diffusion from the interior. The drying of sheet-like materials is essentially a process of surface evaporation. Note that with porous materials, evaporation may occur within the solid. In a porous material that is characterized by pores of diverse sizes, the movement of water may be controlled by capillarity, and not by concentration gradients.

To a large extent the phenomenon of surface evaporation is a diffusion of water vapor from the surface of the sheet through the air film to the surrounding air, into which further dissemination is affected by convection. The air film is relatively thick when the motion of the gas is slight, but with a high gas velocity past the solid surface, the thickness of this film rapidly decreases, with the film never, however, disappearing. In the case of the evaporation of water from a wetted solid surface into air, the inner layer of the air film in contact with the solid is maintained saturated with moisture as long as the moisture concentration of the surface of the sheet is sufficient. According to the law of diffusion, the rate of vapor diffusion (weight per unit of time) varies directly as the cross-sectional area of path taken at right angles to the direction in which the gas is diffusing, directly as the difference of the partial pressure of the vapor at the two points in question, and inversely as the length of path. The thickness of the air film is not known, it is, however, a function of air velocity, since thickness decreases with increasing velocity. Diffusion therefore increases as the velocity goes up. The evaporation of water is accompanied by a definite and large absorption of heat. This heat supply must, in general, come from the surrounding air by conduction through the air film, and is proportional to the temperature difference. As long as the sheet remains sufficiently wet, it assumes the wet-bulb temperature of the air, so that, for given conditions of the drying air, the driving force causing diffusion remains constant. As the water content of the sheet decreases, the concentration of water on the surface eventually becomes so small that the rate of evaporation decreases, and hence the temperature on the surface of the sheet starts to increase. Since the rate of drying is proportional to the difference between the vapor pressure of the liquid and its partial pressure in the surrounding space, material which is appreciably soluble in the liquid with a

resultant vapor pressure lowering, will dry more slowly compared to one which is insoluble. Wet sand as an example will dry more rapidly than a salt at the same moisture level, with all other factors being equivalent.

There are some materials that will retain appreciable amounts of moisture when exposed indefinitely to air of a given temperature and humidity. Usually these materials are hydroscopic in nature; i.e., they tend to absorb moisture. These materials pose a true dynamic equilibrium. The percent equilibrium moisture for a given percent relative humidity always decreases as the air temperature rises with such materials. At the percent relative humidities normally encountered, the percentage of absorbed water tends to be significant for many types of organic materials, such as wood, leather, paper, flour. Only the moisture in excess of that absorbed, namely the total minus the equilibrium moisture will be removed. To achieve greater degrees of drying, high temperature or very dry air is needed.

Drying Equipment

As already noted, drying is a major unit operation, or might be considered a unit process. The hardware used varies with the products, its physical qualities, degree of wetness, and value. Certain materials may be treated directly with fire gases without damage; others, which are more delicate, must be treated indirectly with heat or warm air. Drying with a vacuum permits the maintenance of lower temperatures, but it is more costly than atmospheric drying. Each group of materials has its own class of driers available at its disposal. Liquids containing dissolved solids and thin slurries carrying suspended matter are usually handled in drum or spray driers. Sludge-like materials and pastes may be mixed with dried materials until they crumble and then are dried as solids. Wet solids are frequently dried in rotary driers and cylinders dried lengthwise, through which a current of heated air or hot flue gases travels.

Compartment driers are closets or even rooms in which the material is spread on trays or in shallow pans. The operation is often discontinuous; in order to make it continuous, the pans may be placed on trucks traveling on rails, and these are pushed through a tunnel in which warm air circulates. Belt driers and chain driers have similar goals.

The drying a chemical substance is not a simple process. Drying a mass of finely divided solid particles carrying 30 to 40% water, for example; the rate of evaporation is constant and high as long as the surfaces exposed are wet. After the surface is dry, the water in the interstices must make its way to the surface, a process of diffusion that is slower than evaporation from a wet surface; the rate will then drop. This second part of the process must be modified according to the case with which the material crumbles as it dries, exposing new surfaces.

Drying is concerned with heat transfer, namely - latent heat of vaporization and sensible heat to the vapor molecules, to the water in the pores, and to the solid and material transfer; mainly the transfer of water from the inner portions of the paste or crystal mass to the surface.

The process of drying a thin slurry is essentially a single step: the vaporization of the carrying water, or other liquid, and air in motion or other gas, in order to carry away the vapor and avoid reaching the saturation point at which vaporization would stop. The task is eased by application of a vacuum, which permits an equivalent amount of vapor to develop at a lower temperature. One common means of drying is air drying, which may be combined with draining. The removal of part of the water in the material by application of pressure is another type of drying.

Driers may be divided into two main groups; the first group comprises those machines in which the material is in direct contact with the heating medium; the second group includes those driers in which material is separated from the heating medium by a wall. The heating medium for the first group may be hot air or flue gas; for the second group the medium may be steam, hot water, or flue gas. Each group may be subdivided into driers for liquids and for solids, and those for solids into a drier with or without agitation.

The main cost in drying operations is for energy in the form of heat. In all driers with direct contact between the heating medium and material, there is considerable heat loss, namely, all the heat that was required to raise the gases from room temperature to the temperature at which they are discharged from the drier. The efforts to reduce this lost heat to recirculation and to reheating of the exhaust gas. When flue gases can be used, losses are smaller, and, compared to steam, the cost of raising the latter is eliminated so that direct-heat driers are economical. A brief description of commonly used equipment follows.

Spray Driers: A spray drier (refer to Figure 29) is a piece of equipment that consist of a closed chamber in which a liquid carrying dissolved solids or a thin slurry carrying solids in suspension is atomized. A current of hot air meets the droplets and travels with them cocurrent or past them countercurrent, simultaneously vaporizing the water so that when the bottom, or the discharge point, is reached, only a dry solid remains in fine suspension in an air laden with water vapor. The flow of air may be parallel to the flow of the droplets and downward, which permits complete drying of the material by the time it reaches the bottom. The dried particles are light enough that they follow the air currents. Dried product accumulates in the bottom of an inverted cone, for example, and is pulled away by an exhauster, if not by the escaping air itself, which delivers it to a collector. The air, with all its moisture, travels the same path and is discharged from the collector into the atmosphere. When the flow of air is counter to that of the material, it is essential that the air be made to traverse a curtain of atomized liquid in a wet scrubber, which then retains the entrained particles of dry matter. The spray drier

consists of a drying chamber, an atomizing device, means for moving and heating the air or other gases, and a means for recovering the product. The drying chamber may be 10 to 20 ft in diameter and 15 to 30 ft in height; it may be in the form of a cone or an upright cylinder (refer to Figure 30).
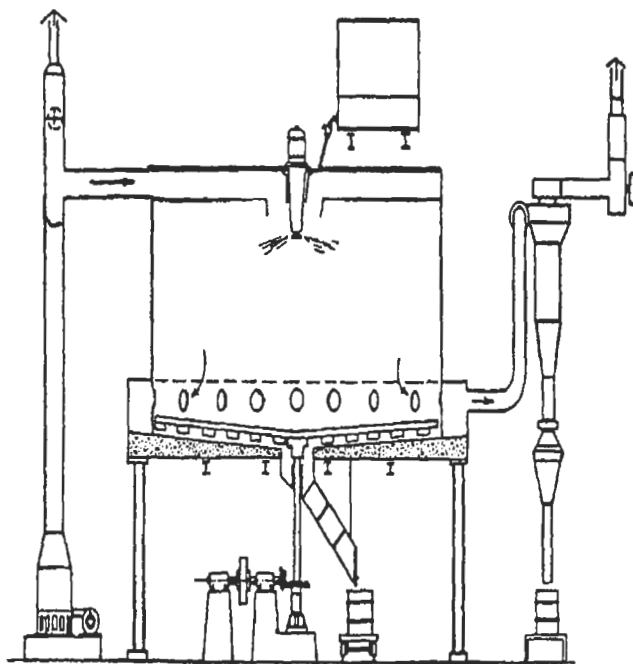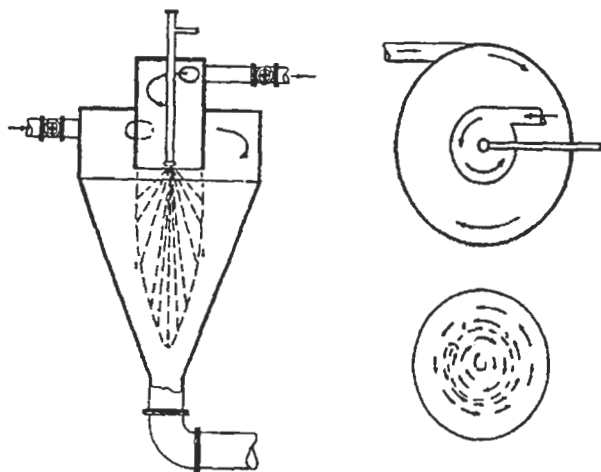

Figure 29. *Shows a spray drier assembly.*

Figure 30. *Cross-sectional view of a double-vortex spray chamber.*

The air is heated by passing it over a nest of finned or grilled tubes in which steam circulates. Flue gases are also used as a source of heat, with a similar arrangement of tubes, except that it is now hot flue gases that pass inside them or mixed air with flue gases from an oil or gas burner (known as a *direct application*). Steam tubes give an air temperature of 300 °F or so; flue gases, indirectly or directly applied, give 500 °F and higher, as high as 1,200 °F. It is desirable to have a high inlet temperature because the amount of the applied heat transferred to the wet material is then greater, as already suggested under heat economy. The direct use of flue gas is not only more economical, but it has the further merit of reducing the danger of dust explosions because of the lower oxygen content of the gas. The temperature that may be used depends on the sensitivity of the material. If it is unstable, a more moderate temperature may be selected. In driers in which hot air travels parallel with the droplets, the hot air comes in contact only with the very wet material, which is protected by its water. As evaporation proceeds, the temperature of the air falls, and it must also be remembered that the droplet is always cooler than the air surrounding it. As the droplet turns to a semisolid and then to a solid, the temperature of the air has dropped so far that there is little chance of scorching the product. The total time the droplet spends in the system, from liquid to collected powder, is between 15 and 30 seconds. The period of active evaporation is probably only about 4 seconds. The spray drier is an instantaneous drier and is particularly well adapted to drying heat-sensitive substances.

The spray drier gives the product a powdery quality; certain materials on spray drying form hollow spheres. The physical quality of the product is likely to determine whether spray drying is to be adopted, irrespective of heat consumption. The shape of the product is greatly influenced by the type of nozzle and with certain materials by the discharge speed of the atomizer. The advantages of spray drying are continuous operation; a short period of contact, which is well indicated by speaking of the spray drier as the instantaneous drier; and the safe handling of heat-sensitive materials.

Flash Driers: The flash drier represents the application of the principle of the spray drier to materials that are solid or semisolid in the wet state. Refer to Figure 31 for an example of a system. The wet material is dried while suspended in finely divided form in a current of heated air. This is accomplished by dropping the wet material into a high-temperature air stream that carries it to a hammer mill or high-speed agitator where the exposed surface is increased. The fine particles leave the mill through a duct small enough in area to maintain the carrying velocities and reach a cyclone separator. The flash drier is an example of parallel-flow operation. A solid particle takes 6 to 8 seconds to pass from the point of entry into the air stream to the collector. The air temperature drops from 1,200 ° to 600 °F, for example, in 2 seconds, or from 1,200 °F to 350 °F in 4 seconds. The time for drying is essentially 2 to 4 seconds, hence the term *flash*. The material itself is not raised in temperature by more than perhaps 100 °F; thus, materials that would burn at the

temperature of the heating agents pass through unharmed. A material having an initial moisture content of 80 % may be reduced to 5 or 6 % in the dried product.

Drum Driers: In drum drying, a liquid containing dissolved solids or a slurry carrying suspended solids is spread on the surface of a large drum lying on its side and heated internally. In the simplest arrangement, the drum dips in a pan underneath and there receives its coat. As the drum revolves, the liquid is dually vaporized, so that after seven-eighths of a revolution, a dried deposit can be scraped off (sometimes loosening is sufficient) by a flexible, adjustable knife. The rotation of the drum is made slow enough so that all of the liquid portions are evaporated as the residual solid approaches the knife. The solid collects on an apron in front of the knife and rolls to a container or to a screw conveyor. The operation of the drum drier, or film drier is continuous. The drum is rotated continuously by a gear driven by a pinion that receives its motion through a belt, a chain, or a reduction gear from a direct-connected motor. The speed of the drum may be regulated by a variable-speed drive to adapt the speed to any slight variation in the liquid. If the material is dry quite a distance before the knife is reached, the speed should be increased; if the material is too wet at the knife, the speed must be decreased. The knife may be held just against the surface, or it may be forced against it by turning the adjusting wheels. The knife supports may be turned through part of a circle so that the angle of the blade of the knife relative to the drum surface may be selected for the greatest shearing effect.
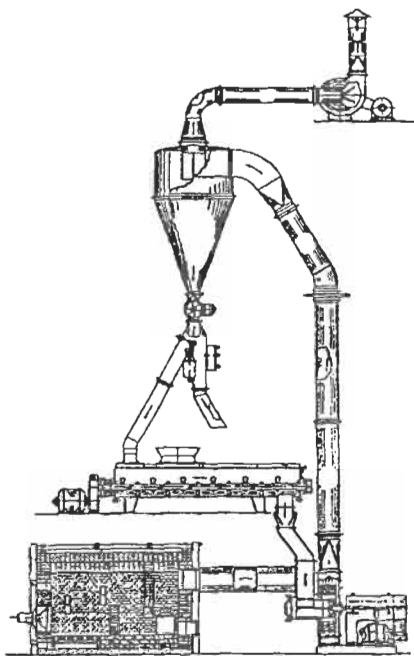
Figure 31. *Flash drying system.*

The drum drier is heated by steam, which enters through the trunnion. The condensate is discharged by means of a scoop or syphon through the second trunnion. Rotation of the drum varies with duty but, in general, will be between 4 and 10 rpm. The drum for the single atmospheric drum drier is a single casting, the face turned true on a lathe and then ground and polished. The materials are cast iron, bronze, or chrome-plated. For dimensions, a drum 5 ft in diameter and 12 ft long is large. Smaller sizes generally are a 24-in. diameter drum, 24 to 36 in. long. Figure 32 shows an atmospheric single-drum drier with dip feed. Besides the knife, a spreader is shown, which regularizes the coating. The heat from the condensing steam passes through the condensate film, through the metal of the drum, and then through the coating on the drum. The maximum rate of evaporation for a dilute solution, which gives up its water more readily than a concentrated one, is as high as 20 lb of water per hour per square foot of drum surface. This, however, only indicates that a high rate of evaporation may be obtained. As the purpose of drum drying is, rather, to produce a quantity of dry material, the true measure of efficiency is the number of pounds of finished product per unit heating surface. The rate of evaporation is determined by the concentration at which the material is fed to the drier. The capacity of a drum drier depends on its dimensions, the speed of rotation (the greater the speed, the greater the discharge, with the reservation expressed below), and the initial concentration of the liquor or slurry; it will depend, furthermore, on the residual moisture allowable in the product, on the heat resistance of the liquor film, on the steam pressure, and on the adhesion of the coat to the surface. The better the adhesion, the heavier is the coating and, therefore, the greater the amount dried in a unit time for a given drier.
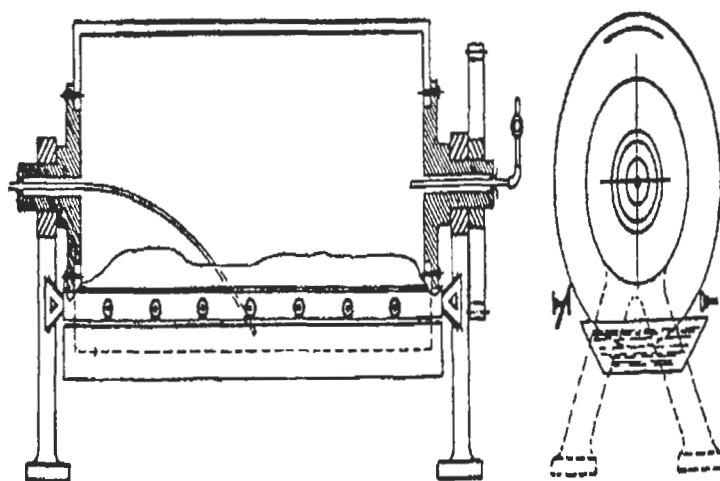
Figure 32. *An atmospheric single drum drier with dip feed.*

The capacity is practically proportional to the surface area. It is slightly favored in the larger sizes because of better mechanical control of operating conditions. The capacity for various products varies greatly, as adherence and liquid film resistance to heat vary over an extremely wide range. The amount of dry product may be from 1-6 lb per square foot of surface per hour. The coating thickness is governed by the clearance between the drums. The larger the clearance, the thicker is the coating and the higher the capacity of the drier. Thee clearance is adjusted by moving the bearings of one of the drums with the adjusting screws to the maximum thickness that can be dried uniformly. The twin-drum drier avoids the dropping of lumps and amount (as high as 25 %) of moisture. The dip feed is adopted for liquors of high concentration, which adhere readily to the hot drum surface. The splash feed, as with the single-drum drier, overcomes the lack of adhesion of many materials to a heated surface. It should be noted that the splash feed liberates the part of the drum surface otherwise buried in the dip pan, so that evaporation takes place from a larger active surface. Finally, the splash feed reduces the concentration of the liquor before its application as a coating. Refer to Figures 33 and 34 for examples of an atmospheric double-drum drier with central feed and an atmospheric twin-drum drier with splash feed, respectively.
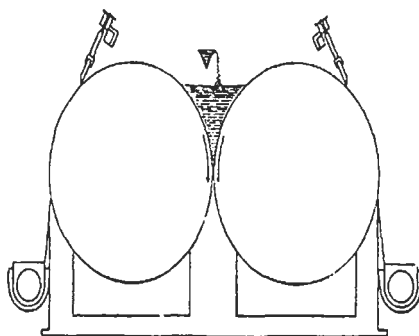
Figure 33. *Atmospheric double-drum drier with center feed.*
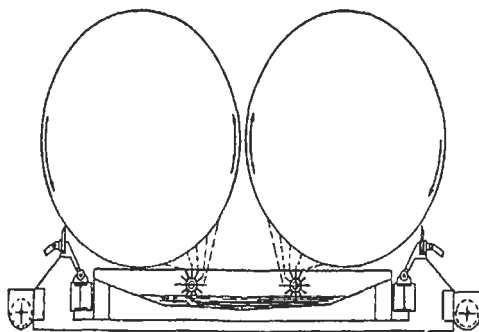
Figure 34. *Atmospheric twin drum drier.*

Rotary Driers: The rotary drier is basically a cylinder, inclined slightly to the horizontal, which may be rotated, or the shell may be stationary, and an agitator may revolve slowly. In either case, the wet material is fed in at the upper end, and the rotation, or agitation, advances the material progressively to the lower end, where it is discharged. Figure 35 illustrates a direct- heat rotary drier. Typical dimensions for a unit like this are 9 ft in diameter and 45 ft in length. In direct-heat, revolving rotary driers, warm air or a mixture of flue gases and air travels through the cylinder. The rate of feed, the speed of rotation or agitation, the volume of heated air or gases, and their temperature are so regulated that the solid is dried just before discharge. The shell fits loosely into a stationary housing at each end. The material is brought to a chute that runs through the housing; the latter also carries the exhaust pipe. The revolving shell runs on two circular tracks and is turned by a gear that meshes with a driven pinion. The inclination is one in sixteen for high capacities and one in thirty for low ones. As the shell revolves, the solid is carried upward one-fourth of the circumference; it then rolls back to a lower level, exposing fresh surfaces to the action of the heat as it does so. Simple revolving rotary driers serve well enough when fuel is cheap. The efficiency is greatly improved by placing longitudinal shelves 3 or 4 in. wide on the inside of the cylinder. Longitudinal shelves are called lifting flights. The shelves carry part of the solid half-way around the circumference and drop it through the whole of a diameter in the central part of the cylinder where the air is hottest and least laden with moisture. By bending the edge of the shelves slightly inward, some of the material is delivered only in the third quarter of the circle, producing a nearly uniform fall of the material throughout the cross section of the cylinder. The heated air streams through a rain of particles. This is the most common form of revolving rotary cylinder. It has great capacity, is simple in operation, and is continuous.
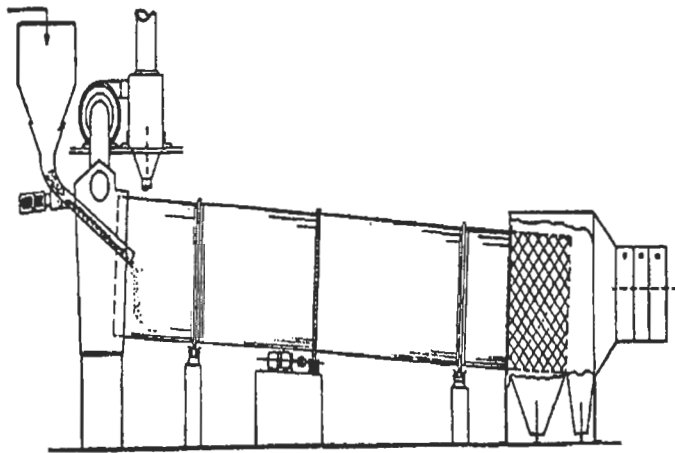
Figure 35. *Direct heat rotary drier.*

The mass of material in the drier is 30 or 40% of the volume of the shell; the air has a narrower channel than the full diameter of the shell, and for the same volume of air the velocity is higher. Velocities are commonly calculated on the basis of the whole shell space. The steam-tube drier has a revolving shell, and the material drops through a bundle of steam tubes, which run the full length of the drier parallel to the shell axis. They are mounted at one end in a steam distributing head, generally in two concentric rows; at the other end, they are loosely supported in a plate, free to expand and vented to remove permanent gases. They may also be mounted on a floating head. Lifting flights are provided when necessary.

Drying Rooms: For drying materials that cannot be agitated, the drying room, and tray drier (which there are many varieties) can be used. In the former, the material may be spread on pans and these stacked in a rack, which leaves a space between the trays for air circulation. The room has loosely fitted side walls of plaster board, or similar material, or even canvas, and contains a steam radiator. The natural circulation of the warm air removes the moisture; the drying period is generally long, for example, one or two days.

Tray, Truck, and Tunnel Driers: In order to accelerate drying, the closet is factory-built with tight walls. It forms a box, and the air is passed by means of a fan over a radiator or over finned tubes and then over the trays. A portion of the air escapes at the discharge opening; the remainder is reheated and recirculated. An amount of new air equivalent to the volume discharged is admitted at the fan. Secondary heating tubes are placed in the path of the air to restore their temperature and heat content. In the tray drier, shallow pans 2 ft by 3 ft by 2 in. deep, for example, are placed on a rack, forming part of the drier. In the truck drier, the rack is on wheels, and the whole may be wheeled in and out of the drier. There may be one or several trucks to each drier, and each truck may have twelve, sixteen or more levels for trays.

A number of tray or truck driers may be set up side by side and air passed through them countercurrent with respect to the ware. The regulation is by means of conduits, by- passes, and dampers. This arrangement is known as a compartment drier. The tunnel drier is a natural extension of the truck drier. It is a long chamber holding a number of trucks that travel in one direction; as a fresh truck is pushed in at one end, by hand or by machinery, the truck with the longest period in the tunnel at the other end is pulled out. The warm air is introduced at the dry end and leaves at or near the wet end. The tunnel drier, then, is composed of a long chamber with doors at both ends, a low-pressure fan, a set of heating tubes or a source of fire gases, and a number of trucks with racks for the trays. There are a number of modifications of such installations designed to the special need they serve.

Belt Driers: A belt drier consists of a set of hinged shelves between two endless chains. As the belt so formed travels on large sprocket wheels, its path lies within

a chamber in which warm gases circulate. The belt is long enough to keep the product within the heated chamber for a number of minutes, perhaps an hour, after which time it is carried to the serving point again. An operator removes each piece and replaces it with a fresh piece. The rate of travel is slow. The purpose of the belt drier is to allow placing the pieces in and taking them out of the drier with a minimum of labor. A belt conveyor drier is a belt drier with the belt made up of stout links with hooks, for example. The belt travels overhead, and the product, let us say flat pieces of plasticized rubber just out of a bath of talc suspension, reaches a long compartment or tunnel in which warm air is circulated. As the belt moves, the suspended rubber approaches the point of discharge, and the time is so computed that it arrives there dry. The material itself also may form a belt, and is done with cloth and with yarn, which is made to travel over rollers through a tunnel-like chamber countercurrent to the air. This type is known as a festoon drier and is used, for example, in making coated paper. The belt may be made of coarse-mesh chain and the material pressed into it in paste form. The belt is made to travel up and down the length of the drying chamber, festoon fashion, or a number of such belts, each one horizontal and a complete loop, may be mounted above one another and made to travel back and forth in opposite directions.

Shelf Driers: The shelf drier has hollow shelves, usually of welded steel, with connections for incoming steam and outgoing drips to each shelf, a number of shelves are superimposed in a square or rectangular box. The material is spread on the shelves directly or on trays that are placed on the shelves, and the heating is through the metal wall of the shelf. In the atmospheric shelf drier an exhauster may be connected to the top of the shell. The rotary shelf drier has stationary, horizontal circular shelves and a rotating vertical central shaft with arms carrying paddles or ploughs. The shelves are hollow, and usually of steel, with steam and drip pipe connections; they have alternating central and circumferential openings, through which the material makes its way downward, traveling over the entire width of each shelf. Sloughs are set to sweep inward or outward, as the shelf may require. At the lowest shelf, the material is discharged at the circumference into barrels or a conveyor. A light steel casing fits around the shelves and permits the removal of gases, which may develop by an exhauster connected to the top of the compartment. Rotary shelf driers are built in all sizes.

Pan Driers: The atmospheric pan drier has a jacketed round pan in which a stirrer or mill revolves slowly, driven from below. The stirrer exposes fresh surfaces and thereby raises the rate of evaporation and, hence, of drying. The dried material is discharged by opening a gate in the side of the pan. The pan drier is a batch machine and is limited to small batches. Pan driers may be used first to evaporate a solution to crystallizing strength and then as a crystallizer by sending cold water instead of steam into the jacket. The effect of the stirrer during crystallization is to prevent the growth of large crystals and to promote, on the contrary, the formation of small, uniform crystals. The mother liquor is then drained off and the crystals

dried in the same apparatus. For the production of "hypo" (sodium thiosulfate) in rice size, for example, the pan drier transformed into a crystallizer gives excellent results. Similarly, it can be used for carrying out a number of operations in succession without transfer of material.

Vacuum Driers: Vacuum driers possess some advantages over atmospheric driers. One is that any danger of contamination by dirt in the drying air is avoided. Another is decomposition or chemical change in the substance by the oxygen of the air. Another is the easy recovery of a valuable vapor such as organic solvents, as well as the lower working temperature. The latter is a controlling factor for certain heat-sensitive materials. The disadvantages of vacuum drying are the first cost of the equipment being greater than operating costs and limited production. When the product is heat-sensitive even to steam temperatures and when the solvent must be recovered, first-cost considerations and efficiency of heat application are overcome.

Miscellaneous Driers: There are a variety of unique drying equipment used in the rubber manufacturing industry for so-called "finishing operations." The term finishing refers to drying of rubber or elastomer products that are manufactured into various product forms such as pellets, crumb, or bales that can be friable, semi-friable or dense. These operations are used in product drying operations where the rubber is made in a solution based process and is often contacted with water at some point to flash off a hydrocarbon diluent phase, leaving behind rubber crumb suspended in a water medium. The flashing stage is normally accomplished in a flash drum, where the "cement" (elastomer suspended in a hydrocarbon diluent such as hexane) is sparged into a hot bath of water in an agitated vessel, or it may be atomized with steam in the flash drum and suspended in a bath of hot water. The elastomer will have a broad particle size distribution and appear popcorn-like. In this state it is referred to as crumb. The solvent is drawn off to an overhead condenser and recycled through the process.

The drying stage then consists of draining the water by transferring the rubber crumb over vibrating screens, and then extruding the rubber through a device called an expeller. The expeller often has several stages to it. By means of shearing and viscous heating, water trapped within the rubber is both squeezed out and flashed off. Care must be taken in this operation so as not to provide excessive shearing and viscous heating. Many elastomers are heat sensitive, and will scorch (i.e., crosslink) or may even combust. For this reason, such products are normally dosed with a variety of antioxidants and fire retardant additives.

Once the rubber is at some specified level of reduced moisture content (say 5 weight percent or less), the elastomer is then shaped into its final product form and packaged. If the product form is a bale, then the next stage of the operation is a baling operation. Usually final stages of drying are done by allowing the product to air dry in the warehouse before being sold (no customer wants to pay for water if they are buying a product on a unit weight basis).

Warehouse drying is normally accomplished by providing an intermediate packaging that 'breaths'. In the case of a rubber bale, the packaging may consist of polyethylene perforated film wrap that enables the surface moisture to evaporate through the film wrap and achieve an acceptable moisture spec: (typically less than 0.5 weight percent for products like EPDM or butyl rubber).

Another common device used in the rubber industry is the thin film evaporator. This device is very often used in the manufacture of ultra-low molecular weight elastomers that are used in sealant applications or specialty coatings, and as processing aids in conventional rubber compounding processes. The thin film evaporator described earlier, has found a multitude of other industry applications, including food processing operations.

Freeze-Drying Technology

The process of freeze-drying is a simple one that removes moisture from a frozen material while it remains in a frozen state retaining the object's shape and structure. Also known as lyophilization, freeze-drying is the most natural means of preservation and it produces a result that favors extended storage for natural items, preserving the item's natural beauty. Freeze-drying is ideal for the preservation of floral products, whole animals, foods, drugs and even for the recovery of water damaged items from fires or floods.

Originally introduced in 1813 by William Hyde Wallaston to the Royal Society in London, the procedure was known as sublimation (converting a liquid in a frozen state directly to a gaseous one). A form of freeze-drying also was practiced by the ancient Indians in the high Andes Mountains. Other processes have tried unsuccessfully to compete with the results of freeze-drying. Processes involving chemicals, cryogenics, sterilization, irradiation and dehydration have been attempted by others, but freeze still is regarded as the finest method of capturing the original state of a material. With the development of mechanical refrigeration and vacuum systems during World War II, freeze-drying was developed to assist the storage of human plasma. Currently, scientists, museums, insurance companies, pharmaceutical manufacturers, food producers, florists and taxidermists are the strongest professional sectors employing the use of freeze-drying, technology.

Freeze-drying, like all drying processes, is a method to separate liquid water from a wet solid product or from a solution or dispersion of given concentration. However, the main difference is that the liquid water is separated by solidification (i.e., the formation of ice crystals) and subsequent vacuum sublimation instead of evaporation. This allows a drying at subzero temperatures which can be advantageous in case of heat-sensitive products. There are two general applications

of freeze-drying: (1) long-term preservation or stabilization of perishable products (e.g., food, biologicals, pharmaceuticals etc.), and (2) production of defined porous structures or internal surfaces (e.g., collagen sponges, catalysators, etc.).

Freeze-drying is a 3-step process: (1) In the first step the sample is frozen solid. During this *solidification* step a phase separation occurs. Most of the water present in the sample is converted to ice crystals (containing almost pure water), whereas the unfrozen water together with the solutes form an amorphous, or so called *glassy phase*. This glassy phase is the basis for the preservation, since it includes the valuable product. (2) Under vacuum the water is removed from the frozen sample. Usually, the vacuum-drying step is divided into two parts: *primary drying* means the sublimation of ice crystals. Afterward, only the glassy phase is left, now having a porous structure. During the *secondary drying* the unfrozen water diffuses out of the glassy matrix and is desorbed from its surface. If the sample is sufficiently dry it can be stored at room temperature without any further loss of quality. (3) The *rehydration* means the reconstitution of the original state by adding water or an aqueous solution. The temperature-concentration-diagram in Figure 36 shows these three steps from a different point of view. It is important to keep the sample temperature below the glass transition temperature to ensure product stability. The glass transition temperature itself depends on both water content and material. Therefore, it might be necessary to use certain additives to improve the glass forming properties of a sample.
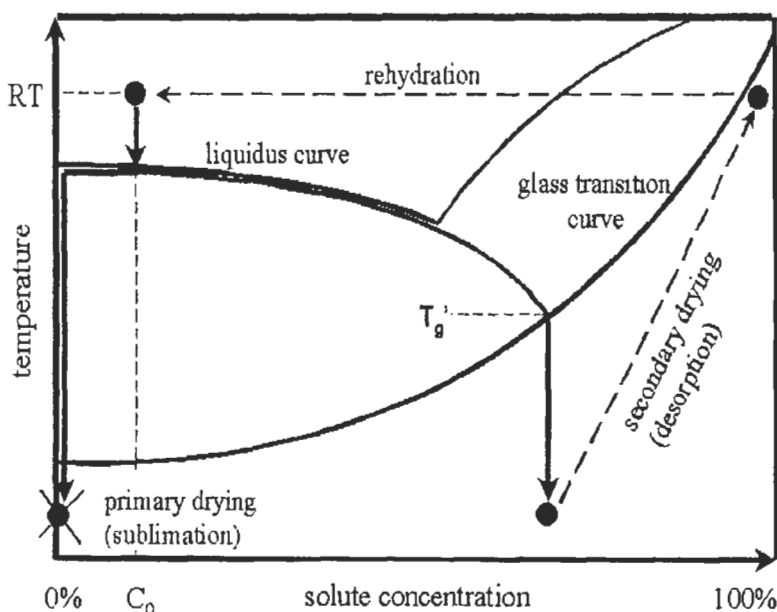
Figure 36. *Temperature versus solute concentration curve.*

Macromolecules (e.g., PVP, HES, Ficoll) as well as sugars (e.g., maltose, lactose, sucrose, trehalose etc.) are often used as stabilizers, since they form a stable glass and have relatively high glass transition temperatures. Freeze-drying is an important process for the long-term preservation of heat-sensitive biological materials. Today, the field of applications contains such topics as relatively simple preservable food, pharmaceutical or biotechnological products, and proliferating bacteria and fungi. The preservation is based on the protective effect of amorphously solidified solutions (glasses) which include the product and prevent ageing. However, the prospects for a successful preservation by freeze-drying decrease with increasing complexity of the biological material. The transition from inanimate to animate material often composes a threshold for the success. Whereas small structural changes may not significantly alter the flavor of food, they are probably lethal in case of a living cell.

Vacuum Drying Technology

A vacuum system typically consists of one or more pumps which are connected to a chamber. The former produces the vacuum, the latter contains whatever apparatus requires the use of the vacuum. In between the two may be various combinations of tubing, fittings and valves. These are required for the system to operate but each introduces other complications such as leaks, additional surface area for outgassing and added resistance to the flow of gas from the chamber to the pumps. Additionally, one or more vacuum gauges are usually connected to the system to monitor pressure.

Traditionally, the pressure in a system is stated in terms of the height of a column of mercury that may be supported by the pressure in the system. At one standard atmosphere the force is 1.03 kg/sq cm (about 14.7 pounds per sq. inch). This pressure will support a mercury column 760 millimeter high (as in a barometer). One millimeter of mercury is the equivalent of 1 Torr. A thousandth of a millimeter is referred to as a micron of mercury or, in more current terminology, 1 milliTorr (mTorr). To be proper in the modern scientific world, the SI system of units is used. Here pressure is referred to in terms of newtons/sq. meter or Pascal (Pa). To convert Torr to Pascal, divide by 0.0075. Table 1 provides conversions between common pressure units. Measurement of pressure in a vacuum system is done with any of a variety of gauges which work through somewhat indirect means e.g. thermal conductivity of the gas or the electrical properties of the gas when ionized. The former are typically used at higher pressures (1 to 1000 mTorr), the latter in lower ranges. Low grade vacuum may be reached using a variety of means. In the range to several 10s of Torr, sealed reciprocating piston compressors (as are commonly found in refrigerators) may be used.

Table 1. *Conversion Factors for Pressure Units*

	mbar	bar	torr	Pa	atm	lb_f-in^{-2}	kg_f-cm^{-2}	in. Hg	mm Hg
1 mbar =	1	10^{-3}	0.75	10^{2}	9.869×10^{-4}	1.45×10^{-2}	1.02×10^{-3}	2.953×10^{-2}	0.75
1 bar =	10^{3}	1	750	10^{5}	0.987	14.5	1.02	29.53	750
1 torr =	1.333	1.333×10^{-3}	1	133.3	0.1316	1.934×10^{-2}	1.36×10^{-3}	3.937×10^{-2}	1
1 Pa =	0.01	1×10^{-5}	7.5×10^{-3}	1	9.87×10^{-6}	1.45×10^{-4}	1.02×10^{-5}	2.953×10^{-4}	7.5×10^{-3}
1 atm =	1013	1.013	760	1.013×10^{5}	1	14.7	1.033	29.92	760
lb_f-in^{-2} =	68.95	6.895×10^{-2}	51.71	6895	6.805×10^{-2}	1	7.03×10^{-2}	2.036	51.7
kg_f-cm^{-2} =	980.7	0.981	735.6	9.807×10^{4}	0.968	14.22	1	28.96	735.6
in. Hg =	33.86	3.386×10^{-2}	25.4	3386	3.342×10^{-2}	0.491	3.453×10^{-2}	1	25.4
mm Hg	1.333	1.333×10^{-3}	1	133.3	1.316×10^{-3}	1.934×10^{-2}	1.36×10^{-3}	3.937×10^{-2}	1

Piston compressors have the disadvantage of the dead space which exists above the piston. This, plus leakage past the piston, limits the degree of vacuum that can be achieved. Better vacuum may be obtained with a rotary, oil sealed pump. This type of pump has a rotating off-center cylindrical rotor that "sweeps" air through the cylindrical housing in which the rotor is located. Air is kept from passing from between the vacuum and pressure sides by means of either a set of two vanes which are arranged across the diameter of the rotor or by means of a sliding single vane mounted in the housing. The entire mechanism of this type of pump is immersed in oil. The oil lubricates the moving parts and also acts as the sealing agent. Single stage rotary compressors, as are used in some air conditioners, are usually good to 1 Torr. (These are typically rather tall and narrow with the wiring at the top of the unit. The inlet is at the bottom/side and the exhaust is at the top. Piston compressors are more squat and, as the internal mechanism is spring mounted, they can be identified by a characteristic 'clunking' sound when shaken.) Air conditioners from GE, Whirlpool, Westinghouse and Sharp commonly use rotary compressors. To get below 1 Torr, a two stage (i.e. one stage in series with another) rotary pump should be used. Some success may be achieved by connecting two rotary air conditioner compressors in series. However, operation may be erratic. There is a type of rotary

compressor which is used for the recharging of refrigeration systems. For less demanding applications, these can offer an economical alternative to industrial grade vacuum pumps.

New, the industrial grade pumps can cost well over $1000. However, a number of suppliers stock rebuilt pumps. In the smaller sizes, fully rebuilt and warranted pumps may be obtained for $500 or so. While the specifications on these industrial pumps will usually state an ultimate vacuum of 0.1 milliTorr, this level of vacuum is usually only attainable under ideal circumstances. A more practical value is 5-10 milliTorr. At lower pressures, what is termed high vacuum, air doesn't respond very well to being squeezed and pushed around by pistons and rotors. At these pressures gas molecules don't really flow. They more or less wander into the pump. The most common type of pump for use in the high vacuum realm is the diffusion pump. This pump, invented by Irving Langmuir in 1916, utilizes a jet of vapor (generated by the boiling of hydrocarbon or synthetic oil) which forces, by momentum transfer, these stray molecules into the high pressure side of the pump. Since these pumps only work at low pressures, the outlet of a diffusion pump must be coupled to a mechanical 'backing' pump. Diffusion pumps are simple, quiet and only require simple (but sometimes tedious) maintenance. The major disadvantages are the backstreaming of oil toward the vacuum chamber (which may be minimized with baffles and or cold traps) and the catastrophic results from accidently opening the system to atmospheric pressure: the oil breaks down and goes everywhere. Mercury was the original pumping fluid. Mercury does not break down and higher forepressures may be tolerated. However, mercury also has a higher vapor pressure and liquid nitrogen cold traps are mandatory to prevent contamination. Oil pumps generally operate at a forepressure in the range of 100 mTorr or less. Ultimate pressures of 0.01 to 0.001 mTorr are readily achievable with small apparatus and simple baffles. Most of today's pumps have 3 stages with inlet sizes ranging from 2 inches on up. Pumping speed is related to the inlet area of the pump. A typical 2 inch pump will have a speed of about 100 liters/sec. For most amateur and small scale laboratory applications, pumps with inlets of 2 to 4 inches are the most convenient and economical to use. A variety of other styles of high vacuum pump have been developed. Such pumps include the turbomolecular (or turbo) pump, which is built roughly like a turbine, and the gas capture pumps (ion, cryoabsorption, and sublimation) which either entrap gas within a material or bury the gas under a constantly deposited film of metal. Most of these pumps are used in applications where extreme cleanliness is required or where very high vacuums need to be attained. However, the turbo is seeing increased use in more common applications.

It is often a requirement for industrial fabrications that once complete they are dried. The reason for this are as diverse as the applications it is used for but typical applications for drying include electrical transformers, gas and liquid pipelines, thermal insulation and cryogenic applications. Drying through the application of vacuum allied with energy input is often the only solution. Drying with vacuum is

effective and complete to very low levels of moisture and does not adversely effect the system that is being dried. Historically oils sealed vacuum pumps have been used as the backing pumps for the drying process. The large water vapor loads encountered, however, cause water to condense in oil sealed pumps, even when using gas ballast, adversely effecting its lifetime, reliability and running costs. To remove problems associated with water condensing in the oil and the decrease in performance seen with oil sealed pumps, modern vacuum technology has introduced a range of dry (oil free) vacuum pumps. Vacuum technology that employs dry pumping is based upon successive pairs of non-contacting pump rotors which transfer and compress process gases from the process pressure to the atmosphere. Dry pumping essentially removes the need for oil in the swept volume and therefore removes the problems normally associated with it. These problems include dust collection in the oil forming an abrasive paste, limited water vapor pumping, reduction in performance over time, extensive maintenance and intervention. One type of dry mechanism that is used in vacuum technology is the claw mechanism. The claw mechanism is so named because the contra-rotating rotors have a claw shaped protrusion. Unlike the Roots mechanism as used in the EH mechanical booster pumps, the claw mechanism pump stage is a true compressor which can handle higher pressure differences between the inlet and outlet ports. As the rotors turn, shown in Figure 37, gas from the vacuum system is drawn in through an inlet port (behind rotor 1 in Figure 37) to fill the cavity in one of the rotors. Further rotation of the claws closes the pump inlet. The trapped gas is then compressed until rotor 2 turns to expose the pump outlet port. The compressed gas is then exhausted. The small volume of gas trapped between the two rotors at the end of the operation cycle is carried over to the next pumping cycle. For the claw mechanism to be used for higher pressure differences between inlet and outlet pressures, several claw stages are required. The rotors for each stage are mounted on a single shaft and housed within a common case. Three such stages will enable the pump to exhaust to atmospheric pressure from an inlet pressure of about 0. 1 mbar.
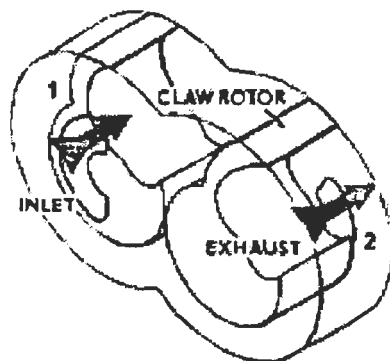
Figure 37. *Claw mechanism.*

Additional information on vacuum technology and its application to drying can be obtained from the American Vacuum Society. The American Vacuum Society (AVS) is a nonprofit organization which promotes communication, dissemination of knowledge, recommended practices, research, and education in the use of vacuum and other controlled environments to develop new materials, process technology, devices, and related understanding of material properties. The AVS is comprised of 8 technical divisions, 4 technical groups, 20 local-area chapters and about 6000 members worldwide. The Society provides stimulating symposia, short courses, and educational outreach both at the national and local levels. The AVS Headquarters is located at 120 Wall Street, 32nd Floor, New York, NY 10005.Phone:212-248-0200; Fax:212-248-0245. Also, the following is a partial list of vacuum equipment and material suppliers which may be contacted for further information:

- Mississauga, Ontario, Canada L5Y 3Y1 (Telephone:905-507-8825; Facsimile: 905-507-8275).

- BOC Edwards: BOC Edwards Vacuum Technology 5975 Falbourne street, Unit 2 Mississauga, Ontario L5R 3w6 (Telephone: 905 501 1223; Facsimile: 905 501 1225).

- Burkholder & Associates: 42 Daniel Avenue Kitchener, Ontario, N2K 1E9, Canada (Telephone: 888-607-0040; Facsimile: 519-570-3354: e-mail: info@ba.ca).

- Kurt J Lesker Canada Inc., 15 Atlantic Avenue Toronto, Ontario M6K 3E7 (Telephone: 416 588 2610; Facsimile: 416 588 2602).

- Leybold Canada Inc., 7050 Telford Way, Unit 5 Mississauga, CDN - Ontario L5S 1v7 (Telephone: 905-6727704; Facsimile: 905-6722249; e-mail:reachus@leybold.on.ca).

- MKS Instruments Canada, Ltd., 30 Concourse Gate Nepean, Ontario, Canada K2E 7v7 (Telephone: 800-267-3551).

- Quantum Technology Corporation, 1370 Alpha Lake Road, Unit 15, Whistler, Bc Canada von Al (Telephone: (604) 938-0030; Faxcsimile: (604) 938-0061).

- Varian Canada Inc., 6705 Millcreek Drive, Unit 5, Mississauga,ontario L5N 5R9 (Telephone: 800-663-2727).

Specialty Industrial Dryers and Applications

There are a variety of specialty dryers tailored to different process applications. Airknife drying is one example of a simple system that has found numerous niche applications. Designs are engineered profile plenum assemblies in lengths typically

ranging from 1 to 144 inches (25.4 to 3,657.6 mm). Some systems employ as many as six airknife assemblies that are powered by a single centrifugal blower to deliver air velocities up to 35,000 fpm. High impact air velocity drying systems are used in any type of canning and bottling plant. Applicable to single or multiple row lines, up to 700 bottles per minute and cans to 1500 per minute. A typical system consists of highly efficient, hard anodized airknives with a 7.5 to 10 hp air blower for use during multiple drying phases. Other applications include parts drying such as machined parts, PC boards, food packaging, produce, glass sheet, rolled steel, plastic, sheet/film, pharmaceuticals, textiles, fabricated parts. Modified designs have proven highly effective in continuous drying of extruded shapes with throughput rates from 1 to 1,500 fpm (0,005 to 15 m/sec). Typical applications include wire and cable, rubber hose, plastic pipe, and a variety of extruded shapes.

High velocity ball dryers and pneumatic conveyor systems are designed to accommodate drying virtually any spherical object on a continuous throughput basis. The drying section utilizes multiple high efficiency airknives matched to a high speed blower assembly to completely dry spherical products in a single file at rates of 2 to 100 parts per minute. The airknife velocity together with blower heat rise of 250 to 800 °F completely dries the product within the chamber while maintaining noise levels of less than 85 db. A pneumatic conveyor carries the product from within the system to a delivery point anywhere in the factory for the next step in the manufacturing process. Standard designs will accommodate the drying and air conveying of part diameters from 12mm to 89mm. With airknife velocities of 35,000 fpm and blowers up to 20 hp, systems can be designed to dry objects much larger than standard capacity.

For drying small fabricated parts, a helical drum dryer is often employed. This machine consists of a perforated, rotating stainless steel drum conveyor that employs a high velocity airknife system to provide continuous throughput and 100 % drying of small, fabricated parts.

A dehumidifying dryer is a rotating module with four separate desiccant beds. At any given moment, two of the desiccant beds (A) are used to dry process air, one desiccant bed (B) is being reactivated and one desiccant bed (C) is being cooled. At the end of a specified time interval (chosen to maximize desiccant performance), the module rotates one-quarter turn. One of the "A" quadrants moves to the reactivation stage. At the same time, the "B" quadrant moves to the cool-down stage and the "C" quadrant moves to the process stage. In the process stage, air returns to the dryer from the hopper and passes through bag filters.Such systems are typically used in resin drying applications. After leaving the "A" quadrants, the dehumidified air moves through a heater box which heats it to the required resin drying temperature; then the air is delivered back to the hopper by a high pressure blower. In the reactivation stage, ambient air which has been super-heated is passed through the desiccant in the bed quadrant being regenerated. As the hot air moves through the desiccant, the moisture which was adsorbed earlier in the drying

process is driven off. Finally, in the cooling stage, the desiccant bed is sealed top and bottom for a two hour period to allow for heat dissipation and ultimately increase the adsorbing efficiency of the desiccant.

Use of Desiccants

Silica gel is the most widely used desiccant. It is an amorphous form of silicon dioxide, which is synthetically produced in the form of hard irregular granules (having the appearance of crystals) or hard irregular beads. A microporous structure of interlocking cavities gives a very high surface area (800 square meters per gram). It is this structure that makes silica gel a high capacity desiccant. Water molecules adhere to the gels surface because it exhibits a lower vapor pressure than the surrounding air. When an equilibrium of equal pressure is reached, no more adsorption occurs. Thus the higher the humidity of the surrounding air, the greater the amount of water that is adsorbed before equilibrium is reached. It is in these higher humidity conditions (above 50% relative humidity) that stored or in-transit items, are susceptible to damage. The beauty of silica gel is the physical adsorption of water vapor into its internal pores. There is no chemical reaction, no by products or side effects. Even when saturated with water vapor, silica gel still has the appearance of a dry product, its shape unchanged.

Silica gel has many other properties that recommend it as a desiccant. It will adsorb up to one third of its own weight in water vapor. This adsorption efficiency is approximately 35% greater that typical desiccant clays, making silica gel the preferred choice where weight or efficiency are important factors. It has an almost indefinite shelf life if stored in airtight conditions. It can be regenerated and reused if required. Gently heating silica gel will drive off the adsorbed moisture and leave it ready for reuse. It is a very inert material, it will not normally attack or corrode other materials and with the exception of strong alkalis and hydrofluoric acid is itself resistant to attack. It is non-toxic and non-flammable.

Standard white silica gel is referred to as being non-indicating. With the addition of a small concentration (about 0.5%) of moisture sensitive cobalt chloride indicator, a self-indicating blue silica gel is produced. Both silica gels will adsorb moisture in exactly the same way, but the self-indicating gel literally indicates when it is becoming saturated, by changing color from blue to pink. This is useful in two ways. Firstly a silica gel sachet that has changed to pink gives an immediate warning that it should be replaced. Secondly, when regenerating silica gel, the self-indicating grade will be ready for reuse, when its color has returned from pink back to blue.

Capsilgels are transparent hard capsules containing silicagel. The capsule is made from hard gelatine. It consists of two cylindrical parts, a body with a hemispherical

base and a corresponding cap of the same shape, but shorter. The internal diameter of the cap and the external diameter of the body are virtually equal. They are held together by a pair of circumferential grooves that interlock when the filled body and cap are pressed together. Both non and self-indicating silica gel can be used to fill the capsule.

The aim of using silica gel sachets is to minimize the risk of irreversible damage to items in transit or store caused by condensing water vapor. By adsorbing water molecules, a silica gel sachet will reduce the relative humidity inside a package to levels at which condensation will not occur. Three main moisture sources need to be considered: (1) that from the air contained in the package; (2) that from the packing added inside of the package (e.g,, for cushioning purposes); and (3) that which diffuses through the outer packaging during storage or transit. These factors are considered in British Standard 1133: Section 19: 1968 as:

(a) The area of the moisture barrier: A (in square meters);

(b) The type of moisture barrier: R is the moisture vapor transmission rate of a barrier (in grams per square meter per 24 hours, measured at 90% relative humidity and 38 °C);

(c) The volume of the air inside the barrier: V (in cubic meters of the container);

(d) The weight and type of any packaging material inside the barrier. This is known as the dunnage factor: DF (D is the weight of packing material in grams and F is a factor depending on the type of dunnage and its probable moisture content) F values are 1/5 for timber of moisture content higher than 14 %, 1/8 for felt, carton board and similar general packaging, and 1/10 for plywood and timber with a moisture content less than 14 %;

(e) The length of time protection is required: M (in months);

(f) The type of climate - temperate (average 25 °C and 75% relative humidity) or tropical (average 38 °C and 90% relative humidity).

The following formulae can be used to calculate the amount of silica gel required, where W is the weight of the silica gel required in grams:

For temperate climates:
$$W = 11 \times A \times R \times M + DF$$

For tropical climates:
$$W = 40 \times A \times R \times M + DF$$

For hermetically sealed containers:
$$W = 170 \times V + DF$$

It is difficult to determine exact weights of silica gel required from the above formulae owing to the many variables that have to be considered, some of which are difficult to measure. The formulae can, however, be a useful guide to indicate the amount of silica gel required to give adequate protection. It is often prudent to choose the nearest standard size silica gel sachet above the value that W comes out at. For large values of W, a more effective solution than using a single large sachet, is using a number of smaller sachets spread around the package. This will reduce the relative humidity at a faster rate.

Condensation of water vapor from air will not occur, even when the relative humidity approaches 100 %, providing the air temperature remains constant. Hence the risk of condensation (and therefore corrosion) arises mainly because of large drops in air temperature. In this case the volume of water vapor is too much for the air to hold. Special attention is needed in situations where: (a) variations between daytime and night-time temperatures reach 20 - 30 °C; (b) seasonal temperature variations are large; and (c) temperature variations occur due to the intermittent functioning of encased apparatus such as electrical conductors and resistors, fuses, circuit breakers or relay motors, which release calories when in use. The greatest risk of condensation is in high humidity environments, where a drop of only 5 °C will on average, cause water vapor to condense. As a general rule it is sufficient to maintain the relative humidity of the enclosed air below 30 % in order to avoid the risk of condensation. Molecular sieves are desiccants with differing properties to those of silica gel. With the appearance of small opaque pinkish beads, molecular sieves are synthetically produced, highly porous crystalline metal-alumino silicates. They have many internal cavities that are linked by window openings of precise diameters. It is these diameters (measured in Angstroms) that classify molecular sieves: 3 Å, 4 Å, 5 Å, and 10 Å (also known as 13 X). Adsorption occurs only of molecules with smaller diameters than these cavity openings. Larger molecules will be excluded from adsorption. Preferentially adsorbed are molecules of greater polarity. This makes molecular sieves ideal for adsorption of water from air and liquids, as water molecules are both polar and very small.

Molecular sieves will adsorb water molecules and other contaminants from liquids and gases down to very low levels - often just 1 part per million. Examples of the applications of the differing molecular sieve grades are: *Grade 3 Å* - Dries unsaturated gases and organic liquids (e.g., methanol). Used to remove water from cleaning fluids in ultrasonic baths. *Grade 4 Å* - Is a general dryer of liquids, natural gases. Also an excellent adsorber of carbon dioxide. *Grade 10 Å/13X* - Is used in air prepurification (due to its high water and carbon dioxide adsorption capacity) and also adsorbs sulphur compounds ('sweetens'). It will remove decomposition products following the quenching of arcing in electrical products. Molecular sieves properties as desiccants differ from silica gel in a number of ways: (1) They adsorb water vapor more rapidly than silica gel; (2) They will reduce water vapor to much

lower levels than silica gel, making their use essential when a very dry product or atmosphere is required; and (3) They perform more effectively as moisture adsorbers at higher temperatures (greater than 25 °C) than silica gel does. Molecular sieves of grades 3 Å, 4 Å and 10 Å/13X are stock items. They are subclassified by their bead sizes, typically 1.0 - 2.0 mm, 1.6 - 2.5 mm, 2.0 - 3.0 mm and 2.5 - 5.0 mm.

Activated clay is a desiccant that is ground and processed from the naturally occurring mineral calcium aluminosilicate. A brownish colored granular material, it adsorbs up to 20% of its own weight on and between the layers of its internal structure. Desiccant activated clay is primarily suited to high volume applications, protecting large items in large enclosures such as shipping containers. In comparison with silica gel, activated clays lower adsorbtion capacity means that an activated clay sachet has to contain 1.35 times more material to perform the equivalent job of a silica gel sachet.

Desiccant grade activated alumina is a highly porous form of aluminium oxide. It has the appearance of white chalky beads. Standard stock sizes have 2 - 5 mm or 5 - 8 mm. beads. An adsorbent from liquids and gases it is supplied usually in bulk packs.

CRYSTALLIZATION

Crystallization generally involves the evaporation and subsequent cooling of a solution to the point of supersaturation, whereupon the formation of crystals takes place. Modern technology often focus on the control of crystal size, since product demands frequently are rigorous in this regard. The process of crystallization is often conducted in evaporators. As in the evaporation of salt and in the recovery of salt and glycerin in soap manufacturing, salt separators are used to remove crystallized materials as rapidly as it settles.

A crystallizer is a vessel in which a hot solution of proper concentration is allowed to cool and form crystals. After crystal separation, there remains a mother liquor, which may be run off, and soluble impurities. Crystallizers for inorganic salts that belong to the heavy chemicals class are rectangular boxes without covers, which are made of wood or steel or are lead-lined, and if provided with an opening for running off the mother liquor, dimensions vary. The moisture, or mother liquor, that clings to the crystals can be removed by sending the crystals to centrifuges from which comparatively dry crystals are discharged at the bottom and enter a rotating cylindrical screen to classify them into sizes. A screen with perforations furnishes various sizes of crystals. Stationary crystallizers are often used because of simplicity and economy. Their major disadvantage is that with older systems, much hand labor is required, and another is that several sizes of crystals are formed

while only one size may be saleable; much time is required for the crop of crystals to deposit. A stationary crystallizer is cooled by natural evaporation and by air cooling its walls and bottom. More rapid cooling is available by using cold water in a jacket, with a better heat transfer assured if a scraper is used to keep the walls free from crystals. The jacketed crystallizer may then take the form of a trough in which long-pitched ribbon scrapers turn. The product is discharged from the trough as a slurry of crystals and mother liquor, which is sent to the centrifuge; the discharge may be continuous if hot liquor is fed in at the other end of the trough continuously; hence a crystallizer with continuous operation results. If the flow of the liquor must be more rapid than the wall area permits, a second or third deck is provided, and the incompletely crystallized liquor from the first cascades to the second and, further, to the third deck.

A double-pipe crystallizer (refer to Figures 38 and 39) consists of fully jacketed pipes with long-pitch agitators, set up in tiers of three or more, each tier operating in series. The inner pile may be 24 inches in diameter, surrounded by a suitably larger pipe, both roughly 10 ft long; several lengths, generally three, are assembled to form one single horizontal length. Three such lengths are set one above another; the hot liquor fed to the upper one moves along the length of the pipe, drops to the intermediate level, travels through its length, and then drops to the lowest level, where it moves again over its whole length to reach the outlet. The cooling water or brine may be fed countercurrent to the flow of the liquor, or each level may receive fresh cooling medium. If both pipes are cast iron, the outer pipe carries baffles in the annular jacket space, which force the water to travel in a spiral path; when the outer pipe is of cast iron and the inner one of steel, stainless steel, or similar metals, a steel rod is welded to the outer side of the latter pipe to form a spiral and produce the same flow in the cooling water. As the liquor moves along the pipes, crystals form, and a slurry is discharged at the outlet, which may be handled in one of two ways. It may be sent past a restricting valve to a magma tank at a lower level, or a riser may cause it to reach the level of the upper tier, there to be discharged into a magma tank. The agitators are driven by sprockets and chains. The shaft of the agitator is adjustable, and the provision makes this adjustment easy. The adjustment is such that the bottom of the pipe is continuously scraped clean. Refer to Figure 40. The rate of rotation of the agitator varies between 5 and 30 rpm, depending upon the material handled. The operation of the crystallizer is continuous; liquor is fed in at the top inlet, and slurry is collected from the outlet, both continuously. The crystallizer may readily be made a link in a sequence of operations, all continuous. The double-pipe crystallizer has a large cooling surface, nearly double that provided in the trough crystallizer; the whole circle of the pipe is cooled. The cubical content per linear foot is also greater, by 25% than that of a trough crystallizer of the same diameter. Another feature of the double-pipe crystallizer is that the cooling is accomplished entirely by the jacket; no evaporation takes place, so that there is no change in the ratio of solute to

solvent. The composition of the liquor or slurry remaining unchanged may be of importance in certain crystallization operations. The double-pipe crystallizer may be operated under pressure. The jacketed crystallizer may also have the form of a circular pan in which a scraper agitator slowly rotates. Water applied to the jacket brings about cooling while the crystallization proceeds. A second way to cool the crystallizer with cold water is by means of one or several stationary coils through which the water circulates, while an agitator keeps the liquor in motion, preventing coating and consequent insulation of the coil walls and bringing new hot layers into contact with cold walls. Cooling is rapid, and the crystals formed are small and uniform. Resulting slurry remains in the vessel until crystallization is complete, when it is discharged to centrifuges or other devices for separating the crystals from the other liquor. Crystallization is more than a matter of cooling a liquor until crystals form. The process varies from one substance to another and even for one substance crystallized from various solvents. Compared to mechanical crystallizers, vacuum crystallizers for equivalent performance are lower in both installation cost and operation cost.

Another major type of equipment among the family of crystallizers is the flaker. The flaker is a piece of equipment that is used for the production by chilling of flakes, chips, large crumbs, or crystals from a hot, concentrated solution or melted crystals.
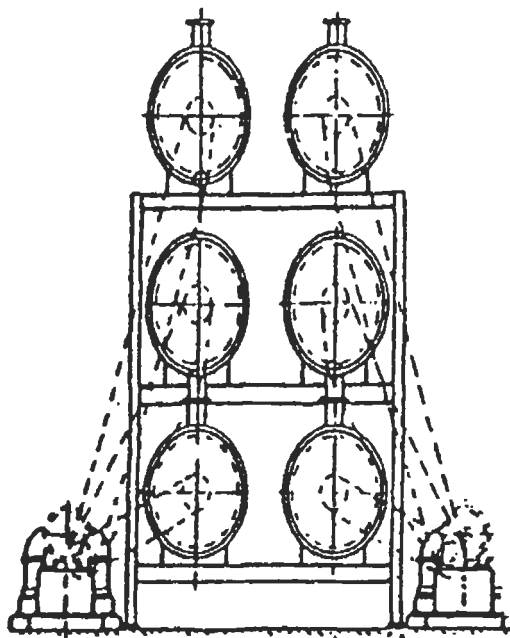
Figure 38. *Front elevation of a double-pipe crystallizer.*
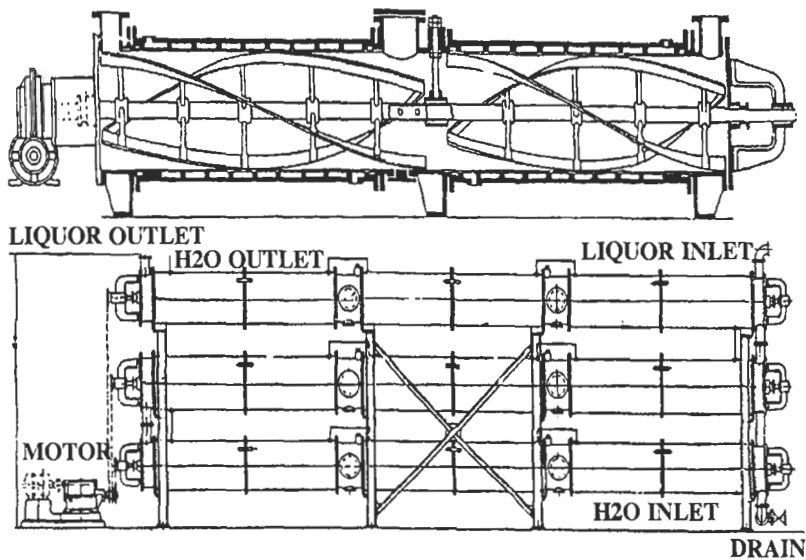
Figure 39. *Vertical cross section of a double-pipe crystallizer showing scraper agitator, adjustable support of shaft bearing, and baffles in the cooling jacket.*
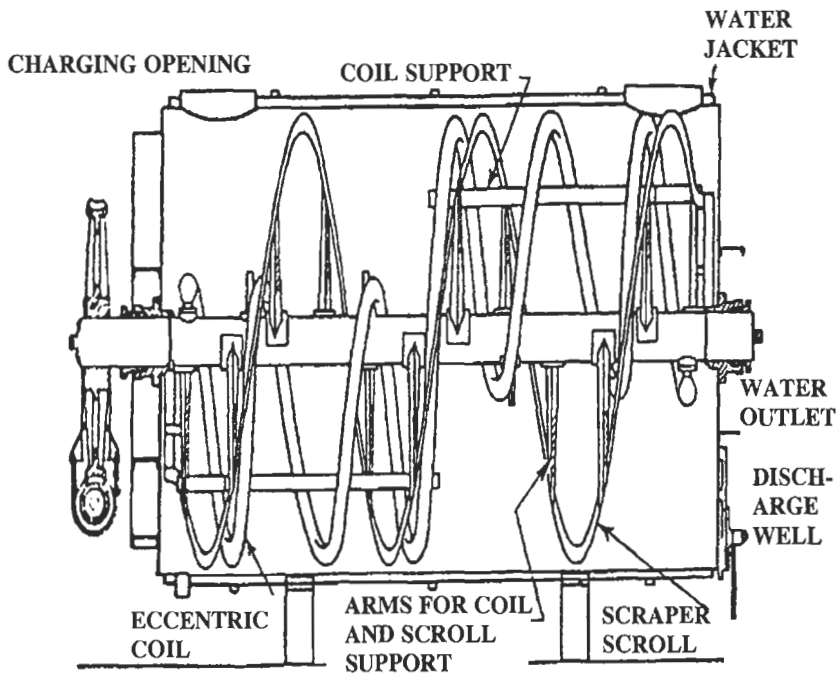
Figure 40. *Crystallizer showing eccentric coil, diamond-shaped arms, and scraper scroll.*

It consists of a hollow, cylindrical metal drum lying on its side; trunnions that permit its rotation; a sturdy, straight, adjustable knife; and a shallow feed pan into which it dips. A large gear mounted on one trunnion is driven by a pinion actuated by motor or pulley. A cast-iron or welded steel frame furnishes the supports. The drum is cooled by water or brine introduced and wasted through the trunnions. A coating of liquid forms on the drum as it dips in the feed pan; as the drum turns, the film at once begins to cool, so that after traveling about three-fourths of the rotation, it is hard and solid. It meets the knife, which scrapes it off, the coating breaking into flakes, chips, or other fragments characteristic of the material. The flakes drop into an apron from which they may be shoveled into shipping drums, or they may drop directly into a screw conveyor that moves them to a chute and packing boxes or barrels. The drum is usually made of a special grade of cast iron with a very smooth surface, which may be chromium-plated. The drum is also made of stainless steel, nickel, or bronze. The motion is generally a steady rotating one, but for certain products it has been found better to move the drum in a series of short jerks. Successful application of the flaker depends upon a low adhesion of the solidified material to the surface of the drum. Should the adhesion be too high, the knife will be unable to lift off the solid and will ride on the material instead of on the drum. The adhesion of some troublesome materials is lessened by a wetting roll, which leaves a film of moisture on the metal just before the coating is formed.

Although specific processes are not covered, an elementary introduction to crystals is provided. Although our best understanding of the atom tells us that the electrons are in constant and rapid motion around the nucleus and the exact location of any electron cannot be known, we can by experimental means determine the most probable locations of electrons and establish energy levels or shells which we can use to help us understand the relations of atoms. In 1913 a Danish scientist, Niels Bohr, developed a model that can be used to understand the way atoms combine into compounds. This model is called the Bohr model and shows a central nucleus with the protons and neutrons either drawn in or simply enumerated. Around this central core are the shells or energy levels. These levels are sometimes referred to as the K, L, M, N, O shells or alter natively, the first, second, third, fourth, fifth, energy levels. Each of these levels has a specific number of electrons it can maximally hold. The degree to which these shells are filled determines how readily it will combine with other elements and in what proportion. Since the shells fill using definite laws and patterns involving energy, they are predictable. Beyond the element calcium, however, the laws become quite complex and need extensive explanation and understanding of subshells. Some periodic tables give the number of electrons in the energy levels as part of the information in the block for each element. By looking at this data, one can come up with partial explanations for the building of these shells. In the absence of such data it is sufficient to declare the numbers 2 and 8 "magic numbers" and let them build the smaller atoms. By drawing a Bohr model of sodium we can see that its K and L shells are filled and

the one remaining electron is by itself in the outer M shell. It will take very little energy to coax that electron away from its atom, leaving only ten electrons with ten negative charges to balance with the eleven protons with their eleven positive charges. When this happens we will have an ion with a $+1$ charge. In the same way if we draw a shell that has seven electrons, one short of that "magic number" eight. In this c ase the chlorine atom would be very happy (anthropomorphically speaking) to grab another electron and fill that shell. In doing so the chlorine atom now would have seventeen protons and eighteen electrons, resulting in a net charge on the atom of -1. This would be a negative ion. When two elements come together in this way we refer to it as an ionic bond. The opposing charges on the two ions cause them to be attracted together. In the case of the oxygen atom, we can see that its outer shell has six electrons, two short of the "magic number" eight. And if we draw a hydrogen atom we can see that it has one electron in its outer shell, half of what it needs to fu lfill the "magic" two count. From this we could deduce that by bringing in another hydrogen atom we can combine the three atoms into H_2O, and each atom's outermost shell would be filled. This is the basis for an understanding of covalent bonds, in which atoms come together and share electrons in their outer shells. The electron clouds overlap and the electrons circle both atoms. The bonds of compounds can influence some substances physical properties. Bonds exist not just between individual atoms but also throughout a crystal. We can look at two forms of the element carbon for an example. Graphite is a slippery black solid, the bonds form sheets of carbon which slide loosely over one another. Diamond on the other hand is a hard, clear crystal with tight tetrahedral bonding that holds the carbon atoms of diamonds securely in place. Further investigation of the importance of chemical bonds can be accomplished by a study of sugar and salt crystals. By comparing melting points and ease of crushing some simple inferences can be made about their bonds. When atoms or molecules are lined up in an orderly arrangement and connected by bonds, and these atoms or molecules have a repeating pattern, we can then say this material is a crystalline substance. The smallest subdivison of a crystal is a unit cell. It is a regular pattern of atoms held together by electrical forces or bonds. These unit cells are far too minute to be seen individually but can be combined together in incredibly large numbers to form visible shapes. As an example of the staggeringly large numbers of unit cells we are talking about we can take as an example sodium chloride, table salt. One typical salt grain has about 5.6 x 10^{18} unit cells. (Each salt unit cell is composed of four atoms of sodium and four atoms of chlorine). When the unit cells group together they leave no empty spaces between themselves. This results in a limited number of crystal systems that can form. These systems can be grouped as follows: (1) Isometric or cubic—three edges of equal length and at right angles to one another; (2) Tetragonal—three edges at right angles but only two edges of equal length; (3) Orthorhombic—three edges at right angles but all edges of different lengths; (4) Monoclinic—two edges at right angle, the other angle not; and all edges of different lengths; (5) Triclinic—all three edges of different lengths and all angles not at right angles; and (6) Hexagonal—two

edges are equal and make angles of 60 to 120 degrees with each other. The third edge is at right angles to them and of different length. Many crystals can be formed by solution, and hence the terms *solute, solvent,* and *solution* are important. The solute is the substance being dissolved and the solvent is the substance doing the dissolving. A solvent can ho ld in solution just so much of the solute. At this point we say the solution is saturated. If there is less solute in the solution than it would ideally hold we would then say it is an unsaturated solution. And in some cases such as when we heat the solvent we can continue to add solute and it will dissolve. When the heat source is removed and the solution's temperature falls the extra solute may remain in solution. This fragile situation is called supersaturation and is the basis for many crystal growth processes. *Solubility,* or the amount of solute which can be dissolved in the solvent, is affected by a number of factors, one of which is the temperature of the solvent. Generally speaking we increase solubility of solid solute when we increase the temperature of the solvent. Crystal growth is a very orderly and regulated process. A crystal grows from the southside with the atoms of the compound being added according to a very specific pattern. If there is not enough space for the crystal to grow unhindered it will increase only until it meets something which gets in its way and then stop. Often many small crystals begin forming at the same time, and they grow until their edges meet at varying angles. They do not join to form a single large crystal but rather remain a jumble of small individual crystals forming a *polycrystalline mass.* The adjoining faces of the crystals are called the *grain boundaries.* These boundaries are particularly evident in metals which have formed by fairly rapid cooling of the molten form. During the cooling process innumerable small crystals form and grow until they bump into a neighboring crystal. Crystals can form from the cooling or evaporation of solutions, or from the cooling of molten solid, or the cooling of vaporized substances.

While the regularity and order of crystals have been stressed thus far, it is important to note that this order can be disturbed. Generally the cause is the inclusion of an impurity. Sometimes this is the result of a crystal forming around a foreign particle. This can usually be detected by microscopic examination. But other times it is actually an invasion by an atom with approximately the same size and shape as the host crystal, and the pattern is not disrupted. This is called a mixed crystal. The classic example of this is alum which is composed of potassium sulfate and aluminum sulfate in a one to one proportion. A similar compound is chrome alum, in which one can find potassium sulfate combined with chromium sulfate. Many crystals in nature demonstrate this mixed crystal condition in the replacement of aluminum by chromium or sometimes iron. Rubies are a good example of this, being composed of aluminum oxide with chromium replacing some of the aluminum, and also sapphires which replace the aluminum with titanium and iron. In some cases a slightly different atomic substance can enter a crystal but only in small quantities. This is called a substitutional impurity. A most relevant example

of this is substitution of phosphorus or boron atoms in silicon crystals. These "impure " compounds are used to make transistors for electronic instruments. Sometimes a different kind of impurity enters a crystal. These foreign atoms may be very small compared to the host substance and fit in between the orderly arranged host atoms. If the host substance has a generous size pattern the invading atoms could be as large as the host atoms themselves. The additional atoms are called interstitial impurities. A well known example of this is carbon and iron, which makes steel. A third kind of defect is called a vacancy. This results from very rapid crystal growth during which some of the atomic sites are simply not filled. The milky or veiled appearance of home-grown crystals, however, is caused by very large openings called voids. It generally occurs when the evaporation of solvent proceeds too rapidly and incomplete crystallization happens. The white coloration is caused by the presence of a liquid solution that is trapped in the open spaces of the crystal. Vacancies on the other hand are far too small to be visible.

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Chapter 4
DISTILLATION EQUIPMENT

INTRODUCTION

Whenever two materials at any temperature possessed a marked difference in their vapor pressures, this difference could be made the basis of a method of separation of the two; and if one or both of the substances were recovered by condensation, the process is called distillation. An equilibrium is established at any temperature between a liquid or a mixture of liquids held in a containing vessel, and the vapor existing above it. Since it is this vapor which ultimately passes out of the containing vessel and when condensed forms the product of the process, the question arises, what is the relation of the composition of this vapor to the composition of the liquid from which it came and with which it is in equilibrium? The answer to this question differs depending on the manufacturing problems of different kinds of mixtures. Obviously, if a volatile component is to be separated from a nonvolatile one, the operation is comparatively simple, and has in principle been described in Chapter 3 under the topic of evaporation. It is necessary only to volatilize the material in one vessel and to condense it in another to accomplish this purpose. In a mechanical mixture of two mutually insoluble liquids sufficiently well agitated to prevent stratification (for example, turpentine and water, heavy fatty acids and water), a dynamic equilibrium is established between the liquid and the vapor when the number of each kind of molecules exiting the liquid is in balance with the number of molecules of each kind again entering the liquid. The pressure existing over the liquid mixture will, therefore, be the sum of these individual pressures, and it follows that a pressure equal to the atmosphere will be reached and boiling will therefore result at a temperature below that at which either of the component liquids would boil alone. This description forms the basis of one type of distillation in which a high-boiling liquid is distilled at a comparatively low temperature by the injection of some low-boiling immiscible liquid or its vapor into the still. In the case where two liquids are mutually soluble, the vapor pressure of each is decreased by the presence of the other, and therefore the sum of their vapor pressures is less than the sum of the vapor pressures of the two liquids before mixing. The composition of the vapor is not independent of the relative amount of the components of the mixture, but is profoundly influenced by each other. This composition can be calculated from the known vapor pressures of the individual pure liquids. When the molecules of two liquids are of relatively the same size, and when there are no

complicated effects when mutually dissolved, such as molecular association, then the composition of the vapor is given by Raoult's law. This law states that that part of the total vapor pressure of a solution of two liquids which is caused by one of the components will equal the product of the vapor pressure of that component in its pure state, and its mole fraction in the liquid. This law, together with Henry's law, are in principle applied in the design of distillation apparatus. In general, the composition of the vapor arising from a solution of one liquid in another is an empirical function of the composition of the solution and must be experimentally determined.

This chapter provides elementary discussions on the subject of distillation, with emphasis given to petroleum refining operations. General terminology and concepts are presented to provide the process engineer with a working knowledge of this important subject, and to become acquainted with the major types of equipment used in distillation operations. Physical properties of hydrocarbon based materials are included in the chapter, as derived from various literature sources and a data base developed by the author over the years as a consultant working in the refining end of the CPI.

OVERVIEW OF DISTILLATION

Distillation is simply defined as a process in which a liquid or vapor mixture of two or more substances is separated into its component fractions of desired purity, by the application and removal of heat. The process is based on the fact that the vapor of a boiling mixture will be richer in the components that have lower boiling points. Hence, when this vapor is cooled and condensed, the condensate will contain more volatile components. At the same time, the original mixture will contain more of the less volatile material. The primary equipment employed in the process of distillation are distillation columns, which are designed to achieve this separation efficiently. Although the layman has a fair idea as to what "distillation" means, the important aspects that seem to be missed from the manufacturing point of view are: (1) distillation is the most common separation technique; (2) it consumes enormous amounts of energy, both in terms of cooling and heating requirements; (3) it can contribute to more than 50% of plant operating costs. The best way to reduce operating costs of existing units, is to improve their efficiency and operation via process optimization and control. To achieve this improvement, a thorough understanding of distillation principles and how distillation systems are designed is essential.

As stated, distillation is the process of heating a liquid until some of its ingredients pass into the vapor phase, and then cooling the vapor to recover it in liquid form by condensation. The main purpose of distillation is to separate a mixture by taking advantage of different substances' readiness to become a vapor. If the difference in boiling points between two substances is great, complete separation may be easily

accomplished by a single-stage distillation. If the boiling points differ only slightly, many redistillations may be required. In the simplest mixture of two mutually soluble liquids with similar chemical structures, the readiness to vaporize of each is undisturbed by the presence of the other. The boiling point of a 50-50 mixture, for example, would be halfway between the boiling points of the pure substances, and the degree of separation achieved by a single distillation would depend only on each substance's readiness to vaporize at this temperature. This simple law was first stated by 19th- century by the French chemist Frangois Marie Raoult (known as *Raoult's law*).

The term "still" is applied only to the vessel in which liquids are boiled during distillation, but the term is sometimes applied to the entire apparatus, including the fractionating column, the condenser, and the receiver in which the distillate is collected. If a water and alcohol distillate is returned from the condenser and made to drip down through a long column onto a series of plates, and if the vapor, as it rises to the condenser, is made to bubble through this liquid at each plate, the vapor and liquid will interact so that some of the water in the vapor condenses and some of the alcohol in the liquid vaporizes. The interaction at each plate is equivalent to a redistillation. This process is referred to by several names in the industry; namely rectification, fractionation, or fractional distillation.

If two insoluble liquids are heated, each is unaffected by the presence of the other and vaporizes to an extent determined only by its own nature. Such a mixture always boils at a temperature lower than is true for either substance alone. This effect may be applied to substances that would be damaged by overheating if distilled in the usual fashion. Substances can also be distilled at temperatures below their normal boiling points by partially evacuating the still. The greater the vacuum, the lower the distillation temperature.

Basic Components of Distillation Columns

There are a variety of configurations for distillation columns, each designed to perform specific types of separations. A simplified way of classifying distillation columns is to look at how they are operated. In this manner, the two major types are batch and continuous columns. In a batch operation, the feed to the column is introduced batch-wise. That is, the column is charged with a 'batch' and then the distillation process is conducted. When the desired separation is achieved, a next batch of feed is introduced. In contrast, continuous columns process a continuous feed stream. No interruptions occur unless there is a problem or upsets with the column or surrounding process units. They are capable of handling high throughputs and are the most common of the two types. The following discussions focus on continuous columns. Continuous columns can be further classified

according to: (1) the nature of the feed that they are processing (*binary column* - feed contains only two components, and *multi-component column* - feed contains more than two components); (2) the number of product streams they have (*multi-product column* - column has more than two product streams); (3) where the extra feed exits when it is used to help with the separation (*extractive distillation* - where the extra feed appears in the bottom product stream , and *azeotropic distillation* - where the extra feed appears at the top product stream); (4) the type of column internals (*tray column* - where trays of various designs are used to hold up the liquid to provide better contact between vapor and liquid, and hence achieve better separation, and the *packed column* - where instead of trays, *packings* are employed to effect contact between vapor and liquid).

There are several important components in a distillation column, each of which is used either to transfer heat energy or enhance mass transfer. The major components in a typical distillation are:

- a vertical shell where the separation of liquid components is carried out,

- column internals such as trays/plates and/or packings which are used to enhance component separations,

- a reboiler to provide the necessary vaporization for the distillation process,

- a condenser to cool and condense the vapor leaving the top of the column,

- a reflux drum to hold the condensed vapor from the top of the column. The liquid (reflux) is recycled back to the column.

The column internals are housed within a vertical shell, and together with the condenser and reboiler, constitute a distillation column. A schematic of a typical distillation unit with a single feed and two product streams is shown in Figure 1.

The liquid mixture that is to be processed is called the feed. The feed is introduced usually somewhere near the middle of the column to a tray known as the feed tray. The feed tray divides the column into a top (enriching or rectification) section and a bottom (stripping) section. The feed flows down the column where it is collected at the bottom in the reboiler. Heat is supplied to the reboiler to generate vapor. The source of heat input can be any suitable fluid, although in most chemical plants this is normally steam. In refineries, the heating source may be the output streams of other columns. The vapor raised in the reboiler is re-introduced into the unit at the bottom of the column. The liquid removed from the reboiler is known as the bottoms product or simply, the *bottoms*. Refer to Figure 2 for a simplified view. The vapor travels up the column, and as it exits the top of the unit, it is cooled by a condenser. The condensed liquid is stored in a holding vessel known as the reflux drum. Some of this liquid is recycled back to the top of the column and this is called the reflux. The condensed liquid that is removed from the system is known as the distillate or top product. Refer to Figure 3 for a simplified view.
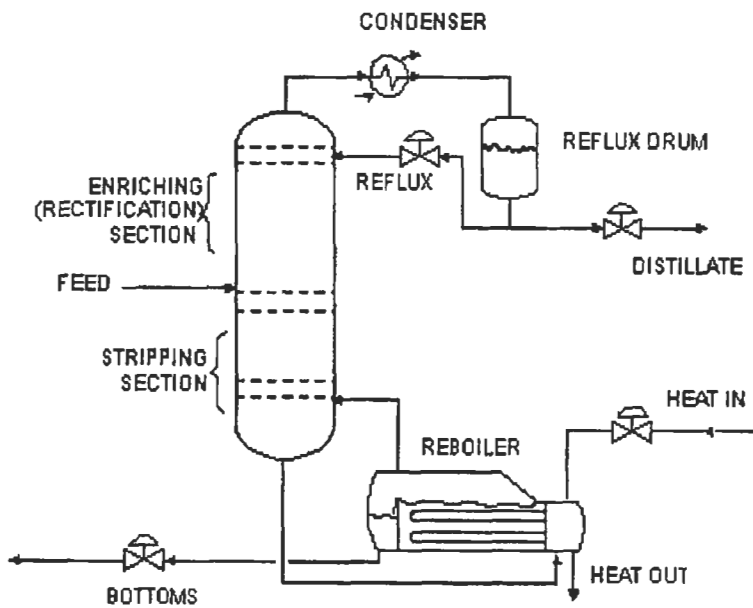
Figure 1. *Basic components of distillation.*
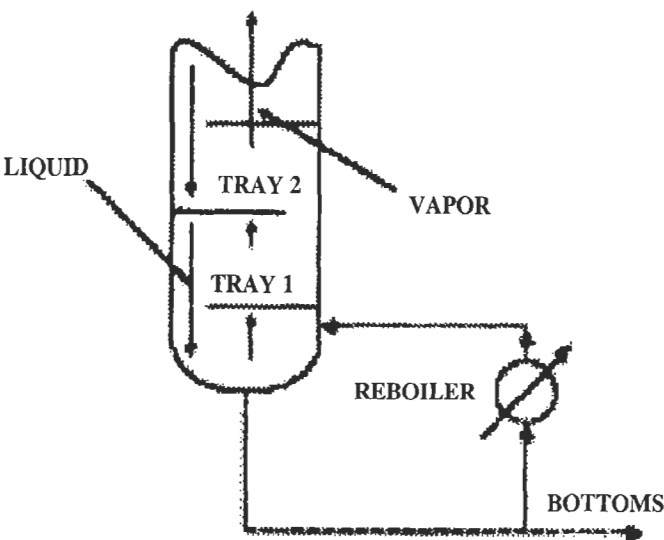
Figure 2. *Diagram shows role of the reboiler.*
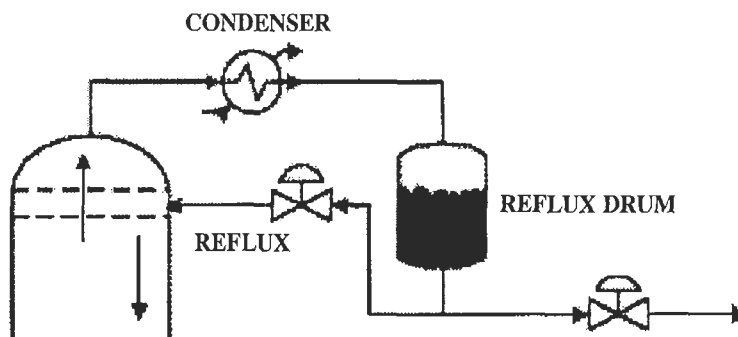
Figure 3. *The role of reflux.*

The terms "trays" and "plates" are used interchangeably. There are many types of tray designs, but the most common ones are bubble cap trays, valve trays, and sieve trays. A bubble cap tray has a riser or chimney fitted over each hole, and a cap that covers the riser. The cap is mounted so that there is a space between riser and cap to allow the passage of vapor. Vapor rises through the chimney and is directed downward by the cap, finally discharging through slots in the cap, and finally bubbling through the liquid on the tray. Bubble-tray or plate towers typically consist of a number of shallow plates or trays over each of which the liquid flows in turn on its way down the tower. The gas enters at the bottom of the tower and is made to flow through a number of bubble caps on each plate. These caps may be of various shapes, though they usually take the form of inverted cups, and their edges are slotted so that the gas escapes from them into the liquid in the form of bubbles. The layout of a typical plate is illustrated Figure 4. The illustration shows the arrangement of bubble caps on a plate together with the risers through which the gas enters the bubble caps and the downcomers which carry the liquid from plate to plate. The inlet weir assists in distributing the liquid over the plate, while the outlet weir maintains the desired depth of liquid. Bubble-plate towers may be preferred to packed towers when: (a) the liquid rate is so low that a packed tower could not be used effectively since the packing would not be adequately wetted; (b) when a difficult distillation duty is required; (c) there is a risk that solid matter may be deposited. Bubble-plate towers, which can be fitted with manholes, are more easily cleaned than packed tower configurations.

In valve trays, perforations are covered by liftable caps. Vapor flows lifts the caps, thus self creating a flow area for the passage of vapor. The lifting cap directs the vapor to flow horizontally into the liquid, thus providing better mixing than is possible in sieve trays.
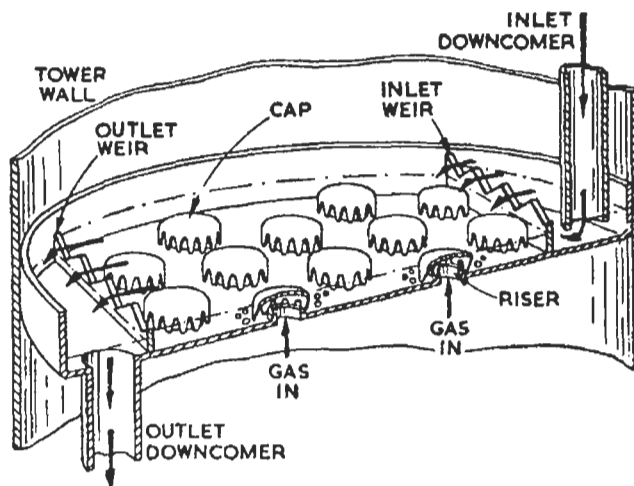
Figure 4. *Bubble cap tray configuration.*

Sieve trays are simply metal plates with holes in them. Vapor passes straight upward through the liquid on the plate. The arrangement, number and size of the holes are design parameters. Because of their efficiency, wide operating range, ease of maintenance and cost factors, sieve and valve trays have replaced the once highly thought of bubble cap trays in many applications.

The flow of liquid and vapor through a tray column is complex. Liquid falls through the downcomers by gravity from one tray to the one below it (refer to Figure 5). A weir on the tray ensures that there is always some liquid (holdup) on the tray and is designed such that the holdup is at a suitable height, e.g., such that the bubble caps are covered by liquid. The vapor flows up the column and is forced to pass through the liquid, via the openings on each tray. The area allowed for the passage of vapor on each tray is called the active tray area. The hotter vapor flows through the liquid on the tray above, and transfers heat to the liquid. During this process some of the vapor condenses adding to the liquid on the tray. The condensate, however, is richer in the less volatile components that is in the vapor. In additionally, because of the heat input from the vapor, the liquid on the tray boils, generating more vapor. This vapor, which moves up to the next tray in the column, is richer in the more volatile components. This continuous and intimate contacting between vapor and liquid occurs on each tray in the column and brings about the separation between low boiling point components and those with higher boiling points. In essence, a tray serves as a mini-column, with each one contributing its share to the overall separation. As such, the more trays there are in a column, the better the degree of separation. Hence, the overall separation efficiency depends significantly on the design of the tray. Trays are designed to maximize vapor-liquid contacting, and hence focus is given to the extent of liquid distribution and vapor distribution achieved by the design.
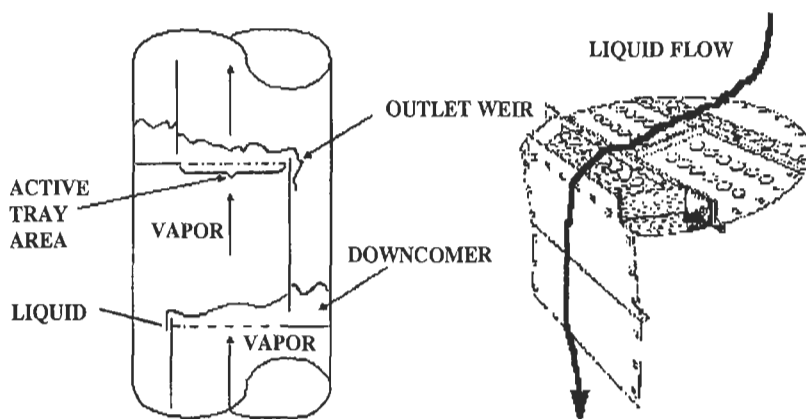
Figure 5. *Downcomer operation and flow over a tray.*

Figure 6. *Sketch of common packing configurations.*

The more intimate the contacting between vapor and liquid, the better the separation that each tray achieves. This means that fewer trays will be needed in order to achieve the same degree of separation. This will result in lower construction costs and energy consumption. Trays alone do not always provide the intimate contact sought. As such, tray designs are sometimes assisted by the addition of packing configurations. Packings are simply passive objects that are designed to increase the interfacial area available for vapor-liquid contacting. Additional discussions on packing materials can be found in Chapter 5, under the section dealing with absorption. Figure 6 illustrates some common geometries of inert packing materials often used in distillation applications. Their role is simply to provide additional surface contact between the vapor and liquid in the column, and to do so without introducing excessive pressure drop across the column. High pressure drop means that more energy is needed to drive the vapor up through a distillation column, and as such there would be higher operating costs. Another very important reason why

inert packing materials are considered is in debottlenecking a column. A tray column that is facing throughput problems can be debottlenecked by replacing a section of trays with packing. The packing will provide additional interfacial contact area for the liquid-vapor contact, thereby increasing the efficiency of the separation for the same column height. In addition, packed columns tend to be shorter than tray-type columns. The packed column is often referred to as a continuous-contact column, whereas a trayed column is called a staged-contact column because of the manner in which the vapor and liquid come into contact.

The function of reboilers has already been discussed. These components are essentially heat exchangers that are used to transfer heat to bring the liquid at the bottom of the column to its boiling point (refer also to discussions in Chapter 1). The principle types employed are jacketted kettles, simple kettle type reboilers, internal reboilers, and thermo-syphon reboilers. Examples of each type are illustrated in Figure 7.
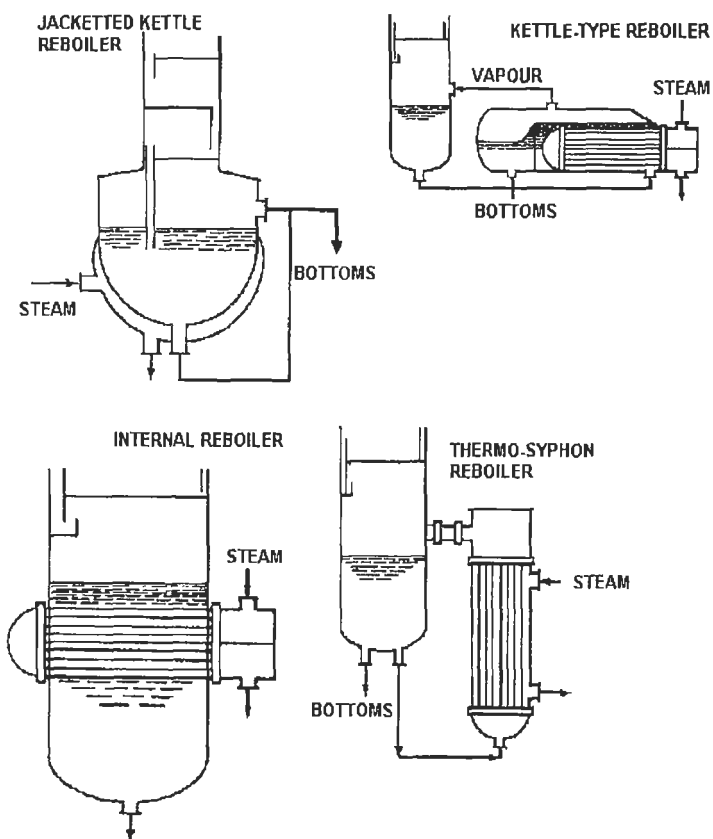
Figure 7. *Common types of reboilers*.

Design Principles

The process of distillation is aimed at the separation of components from a liquid mixture. This process depends on the differences in boiling points of the individual components. Also, depending on the concentrations of the components present, the liquid mixture will have different boiling point characteristics. This means that distillation processes depends on the vapor pressure characteristics of liquid mixtures.

The vapor pressure of a liquid at a particular temperature is the equilibrium pressure exerted by molecules leaving and entering the liquid surface. Some general concepts to recognize about regarding vapor pressure are first, energy input raises the vapor pressure. Also, vapor pressure is related to boiling. A liquid boils when its vapor pressure equals the surrounding pressure. The ease with which a liquid boils depends on its volatility. Liquids with high vapor pressures (i.e., volatile liquids) will boil at lower temperatures. We should also recognize that the vapor pressure and hence the boiling point of a liquid mixture depends on the relative amounts of the components in the mixture. Distillation is accomplished because of the differences in the volatility of the components in a liquid mixture.

It is the boiling point diagram that provides an understanding of the process. The boiling point diagram shows how the equilibrium compositions of the components in a liquid mixture vary with temperature at a fixed pressure. Consider an example of a liquid mixture containing 2 components: A and B. Figure 8 shows the boiling point diagram for this binary mixture. The boiling point of A is that at which the mole fraction of A is unity. The boiling point of B is that at which the mole fraction of A is zero. In this example, A is the more volatile component and therefore has a lower boiling point than B. The upper curve in the diagram is called the dew-point curve while the lower one is called the bubble-point curve. The region above the dew-point curve shows the equilibrium composition of the superheated vapor while the region below the bubble-point curve shows the equilibrium composition of the subcooled liquid. For example, when a subcooled liquid with mole fraction of A = 0.4 (point A) is heated, its concentration remains constant until it reaches the bubble-point (point B), when it starts to boil. The vapors evolved during the boiling has the equilibrium composition given by point C in Figure 8, approximately 0.8 mole fraction A. This is approximately 50% richer in A than the original liquid. This difference between liquid and vapor compositions is the basis for any distillation process.

A term of great importance is relative volatility. Relative volatility is a measure of the differences in volatility between two components, and hence their boiling points. It indicates how easy or difficult a particular separation will be. The relative volatility of component 'i' with, respect to component "j" is defined by the following relationship:

$$\alpha_{ij} = [y_i/x_i] / [y_j/x_j]$$

where y_i is the mole fraction of component "i" in the vapor, and x_i is the mole fraction of component "i" in the liquid. We can conclude that if the relative volatility between two components is very close to one, it is an indication that they have very similar vapor pressure characteristics. This means that they have very similar boiling points and therefore, it will be relatively difficult to separate the two components by means of distillation.

Since the boiling point properties of the components in the mixture being separated are so critical to the distillation process, the vapor-liquid equilibrium (VLE) relationship is of importance. Specifically, it is the VLE data for a mixture which establishes the required height of a column for a desired degree of separation. Constant pressure VLE data is derived from boiling point diagrams, from which a VLE curve can be constructed; like the one illustrated in Figure 9 for a binary mixture. The VLE plot shown expresses the bubble-point and the dew-point of a binary mixture at constant pressure. The curve is called the equilibrium line, and it describes the compositions of the liquid and vapor in equilibrium at a constant pressure condition.
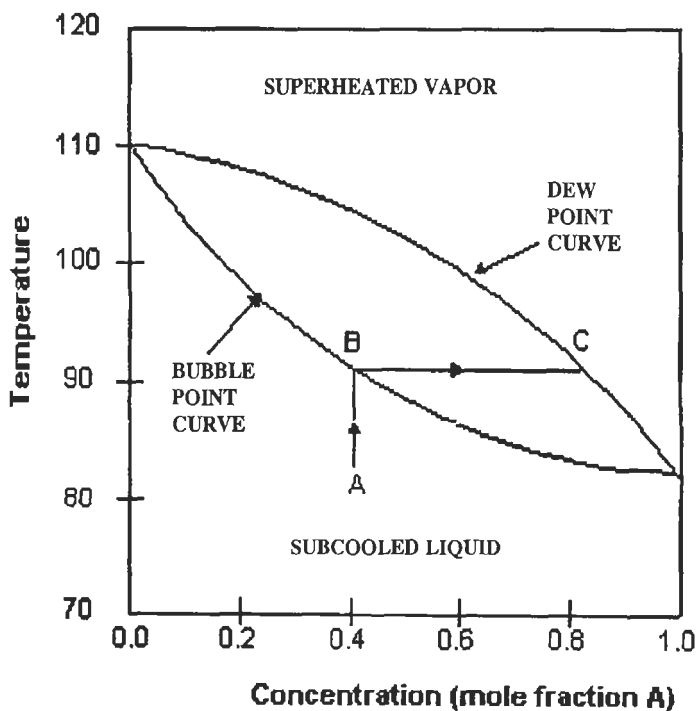
Figure 8. *Boiling point diagram for binary mixture.*
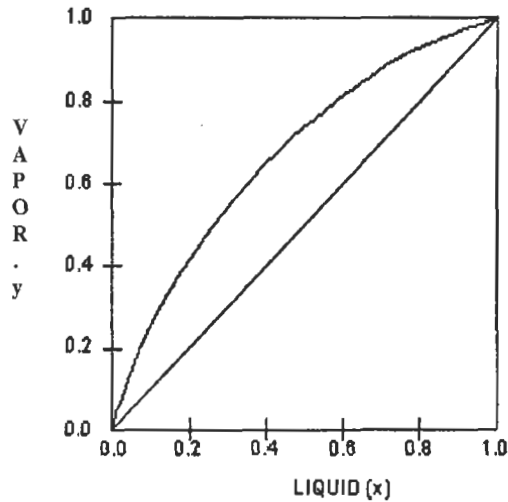
Figure 9. *VLE plot showing equilibrium curve.*
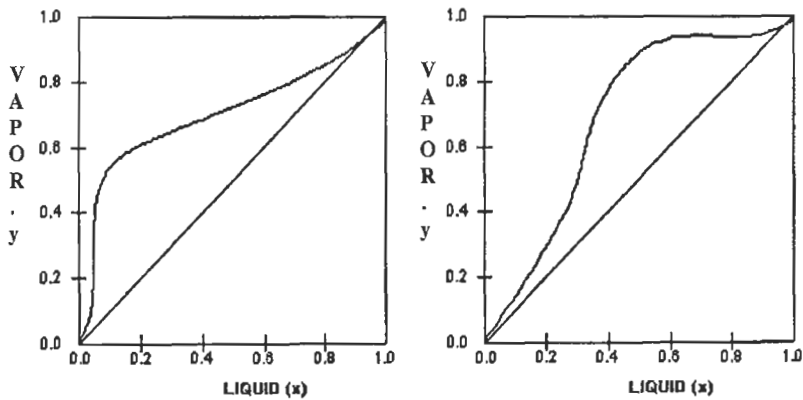
Figure 10. *Examples of non-ideal VLE curves.*

Figure 9 is the VLE plot for a binary mixture that has essentially a uniform equilibrium, and therefore represents a relatively easy separation. However, there are many cases where non-ideal separations are encountered. These more difficult distillations are defined by the examples shown in Figure 10.

An important system in distillation is an azeotropic mixture. An azeotrope is a liquid mixture which when vaporized, produces the same composition as the liquid. The VLE plots illustrated in Figure 11 show two different azeotropic systems: one with a minimum boiling point and one with a maximum boiling point. In both plots, the equilibrium curves cross the diagonal lines.
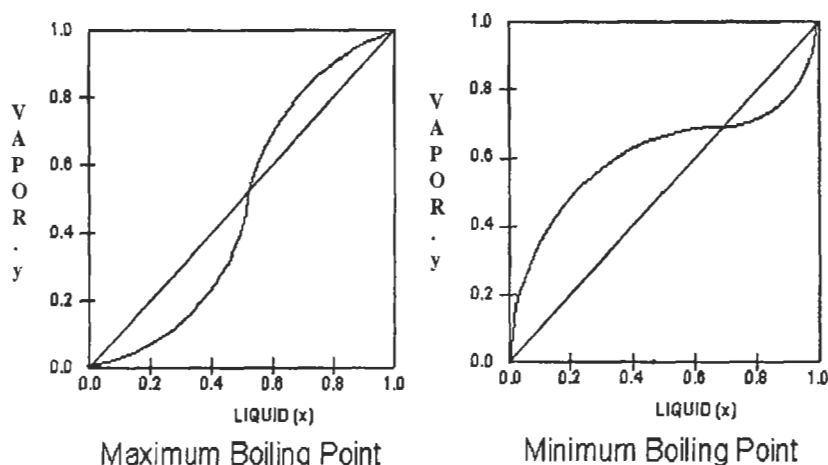
Figure 11. *VLE curves for azeotropic systems.*

These are azeotropic points where the azeotropes occur. In other words, azeotropic systems give rise to VLE plots where the equilibrium curves crosses the diagonals. Both plots are however, obtained from homogenous azeotropic systems. An azeotrope that contains one liquid phase in contact with vapor is called a homogenous azeotrope. A homogenous azeotrope cannot be separated by conventional distillation. However, vacuum distillation may be used as the lower pressures can shift the azeotropic point. Alternatively, an additional substance may added to shift the azeotropic point to a more favorable position. When this additional component appears in appreciable amounts at the top of the column, the operation is referred to as an *azeotropic distillation*. When the additional component appears mostly at the bottom of the column, the operation is called *extractive distillation*.

Another distinction describing a azeotropic system is illustrated in Figure 12. This plot describes the case of a heterogenous azeotrope. Heterogenous azeotropes can be identified by the flat portion on the equilibrium diagram. They may be separated in two distillation columns since these substances usually form two liquid phases with widely differing compositions. The phases may be separated using settling tanks under appropriate conditions.

The design of a distillation column is based on information derived from the VLE diagram describing the mixtures to be separated. The vapor-liquid equilibrium characteristics are indicated by the characteristic shapes of the equilibrium curves. This is what determines the number of stages, and hence the number of trays needed for a separation. Although column designs are often proprietary, the classical method of McCabe-Thiele for binary columns is instructive on the principles of design.
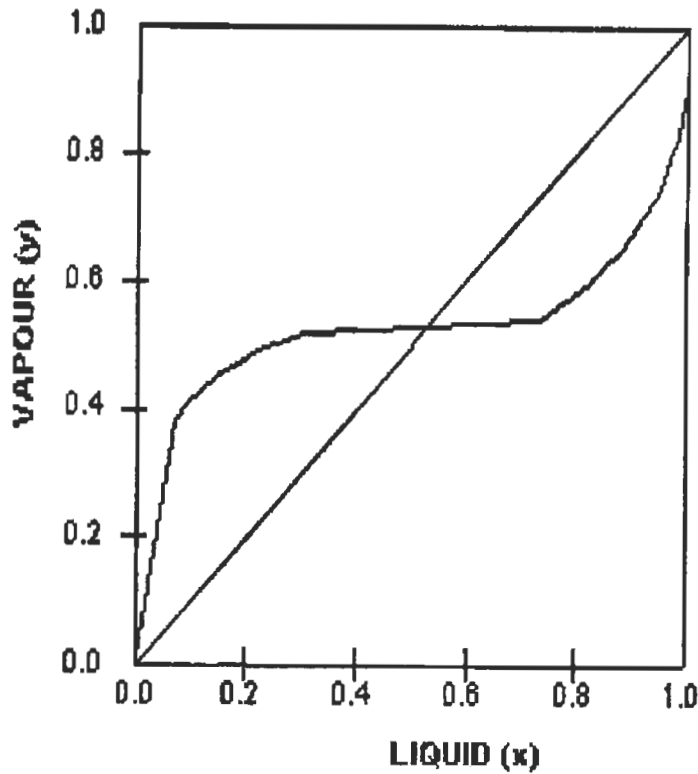
Figure 12. *VLE curve for a heterogeneous azeotropic system.*

McCabe-Thiele is a graphical design that uses the VLE plot to determine the theoretical number of stages required to effect the separation of a binary mixture. It assumes constant molar overflow. This implies that the molal heats of vaporization of the components are roughly the same. In addition, it is assumed that heat effects (heats of solution, heat losses to and from column, etc.) are negligible, and that for every mole of vapor condensed, 1 mole of liquid is vaporized. The design procedure is as follows. Given the VLE diagram of the binary mixture, operating lines are drawn first. The operating lines define the mass balance relationships between the liquid and vapor phases in the column. There is one operating line for the bottom (stripping) section of the column, and one for the top (rectification or enriching) section of the column. Use of the constant molar overflow assumption also ensures that the operating lines are straight lines. The operating line for the rectification section is constructed as follows: First the desired top product composition is located on the VLE diagram, and a vertical line produced until it intersects the diagonal line that splits the VLE plot in half. A line with slope R/(R+1) is then drawn from this intersection point. Refer to Figure 13 for illustration of the procedure.
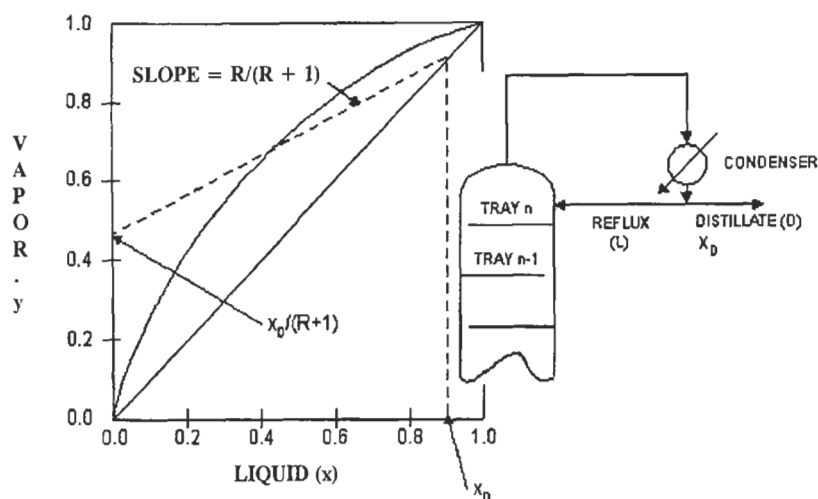
Figure 13. *Application of McCabe-Thiele to VLE diagram.*
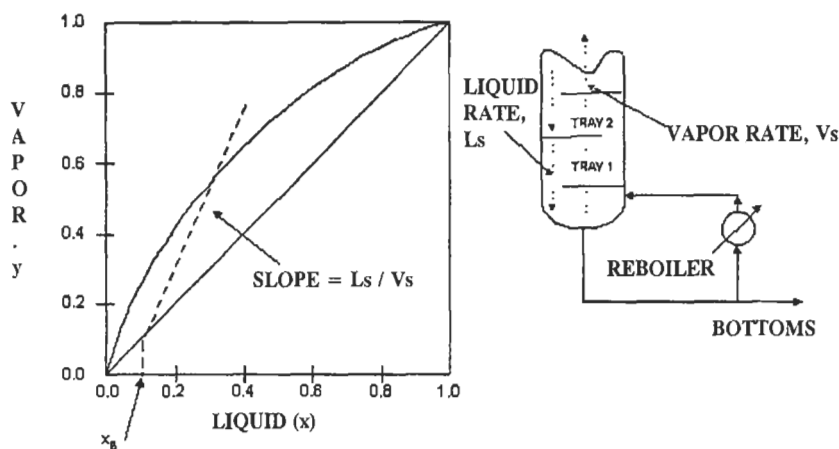
Figure 14. *Construction of operating line for stripping section.*

In Figure 13, R is defined as the ratio of reflux flow (L) to distillate flow (D), and is called the *reflux ratio*. The reflux ratio is a measure of how much of the material is going up through the top of the column and is returned back to the column as reflux. In a similar fashion, the operating line for the stripping section is constructed. In this case the starting point is the desired bottom product composition. A vertical line is drawn from this point to the diagonal line, and a line of slope L_s/V_s is drawn as illustrated in Figure 14. In this figure, L is defined as the liquid rate flowing down the stripping section of the column, while V_s is the vapor

rate of flow up the stripping section of the column. The slope of the operating line for the stripping section is a ratio between the liquid and vapor flows in that part of the column . The McCabe-Thiele method assumes that the liquid on a tray and the vapor above it are in equilibrium. How this is related to the VLE plot and the operating lines is depicted graphically in Figure 15. Figure 15 shows a magnified section of the operating line for the stripping section in relation to the corresponding nth stage in the column. The L's are the liquid flows while the V's represent the vapor flows. Parameters x and y denote liquid and vapor compositions and the subscripts denote the origin of the flows or compositions. The denotation $n - 1$ refers to material from the stage below stage n, while $n + 1$ refers to material from the stage above stage n. The liquid in stage n and the vapor above it are in equilibrium, therefore, x_n and y_n lie on the equilibrium line. Since the vapor is carried to the tray above without changing composition, this is depicted as a horizontal line on the VLE plot. Its intersection with the operating line provides the composition of the liquid on tray $n + 1$, as the operating line defines the material balance on the trays. The composition of the vapor above the $n + 1$ tray is obtained from the intersection of the vertical line from this point to the equilibrium line. By repeatedly applying the graphical construction technique, a number of corner sections are created, with each section being equivalent to a stage of the distillation. This is the basis of sizing distillation columns using the McCabe-Thiele graphical design method.
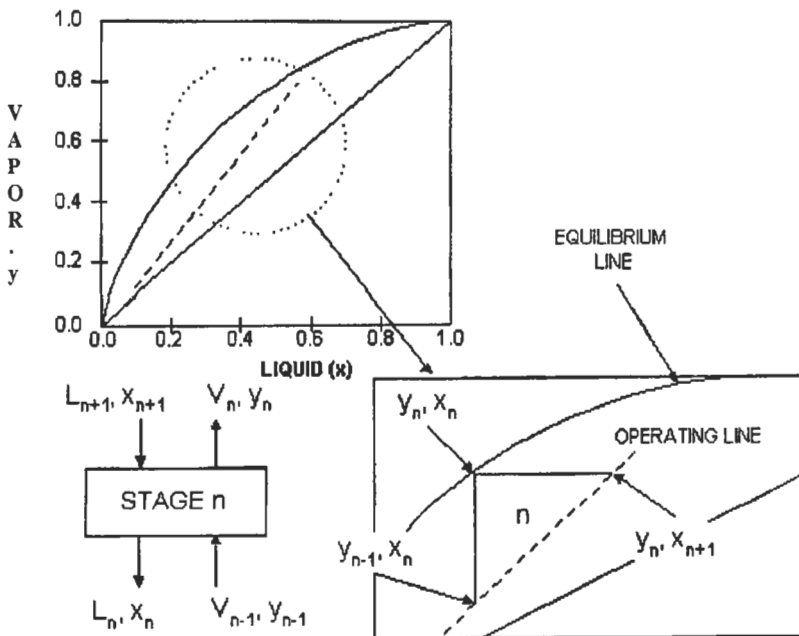
Figure 15. *Illustrates the construction of an operating line.*

From the operating lines for both stripping and rectification sections, the graphical construction described above is further illustrated in Figure 16, which shows that 7 theoretical stages are required to achieve a theoretical separation. The required number of trays (as opposed to stages) is one less than the number of stages since the graphical construction includes the contribution of the reboiler in completing the separation. The actual number of trays required is equal to the ratio of the number of theoretical trays to the tray efficiency. Typical values for tray efficiency ranges from 0.5 to 0.7. Tray efficiency depends on such factors as the type of trays being used, and internal liquid and vapor flow conditions. Sometimes, additional trays are added (up to 10%) to accommodate the possibility that the column may be under-designed. Figure 16 also helps to illustrate that the binary feed should be introduced at the 4th stage. However, if the feed composition is such that it does not coincide with the intersection of the operating lines, this means that the feed is not a saturated liquid. The condition of the feed can be deduced by the slope of the feed line or so-called q-line. The q-line is that line drawn between the intersection of the operating lines, and where the feed composition lies on the diagonal line.
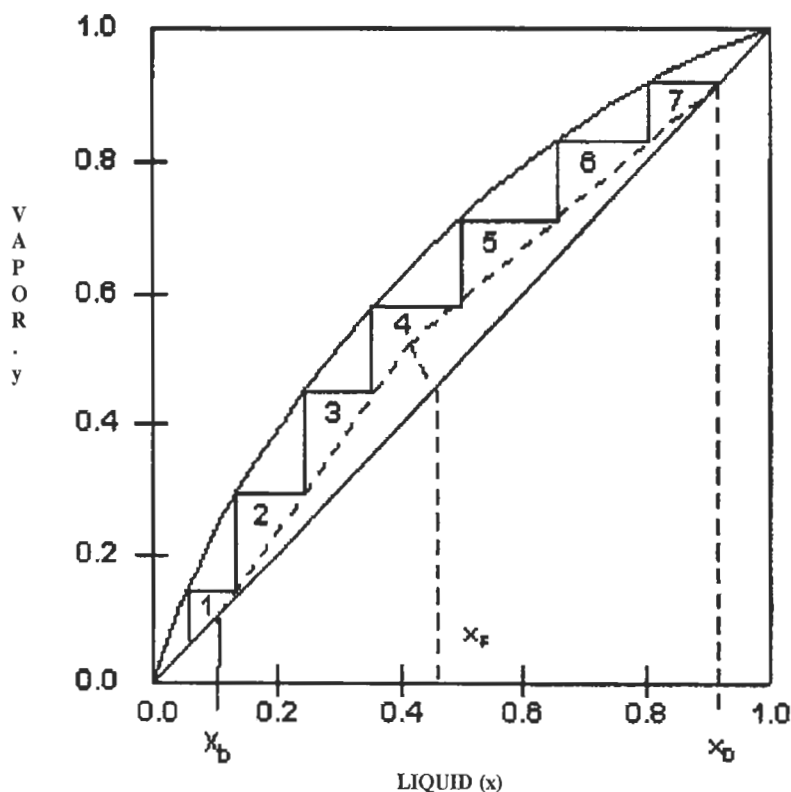
Figure 16. *Example of a seven stage distillation.*

The state of the feed established the slope of the feed line. For examples: saturated vapor exists for $q = 0$; $q = 1$ for saturated liquid; for a mix of liquid and vapor, $0 \leq q \leq 1$; for a subcooled liquid $q \geq 1$; and for a superheated vapor, $q \leq 0$.

From information the feed mixture conditions, the q-line can be constructed and applied in the McCabe-Thiele design. However, excluding the equilibrium line, only two other pairs of lines can be used in the McCabe-Thiele procedure. These are the feed-line and rectification section operating lines, the feed-line and stripping section operating lines, and the stripping and rectification operating lines. The reason for this being that these pairs of lines determine the third.

Determining the number of stages required for the desired degree of separation and the location of the feed tray is only the first step in generating an overall distillation column design. Other factors that need to be considered are tray spacings; column diameter; internal configurations; heating and cooling duties. All of these can lead to conflicting design parameters and trade-offs. Thus, distillation column design is often an iterative procedure. If the conflicts are not resolved at the design stage, then the column will not perform well in practice.

The state of the feed mixture and feed composition not only affects the operating lines and hence the number of stages required for separation, but also the location of the feed tray. During operation, if the deviations from design specifications are excessive, then the column may no longer be able handle the separation task. To overcome the problems associated with the feed, some column are designed to have multiple feed points when the feed is expected to contain varying amounts of components. It is important to note that as the reflux ratio increases, the gradient of operating line for the rectification section moves towards a maximum value of unity. Physically, what this means is that more and more liquid that is rich in the more volatile components are being recycled back into the column. Separation then improves and thus less trays are required to achieve the same degree of separation. Minimum trays are required under total reflux conditions, i.e. there is no withdrawal of distillate. On the other hand, as the reflux decreases, the operating line for the rectification section moves towards the equilibrium line. The 'pinch' between operating and equilibrium lines becomes more pronounced and more and more trays are needed. The McCabe-Thiele method easily verifies this.

The limiting condition occurs at minimum reflux ration, when an infinite number of trays will be required to effect separation. Most columns are designed to operate between 1.2 to 1.5 times the minimum reflux ratio because this is approximately the region of minimum operating costs (more reflux means higher reboiler duty).

A critical consideration in the design of a distillation column is the vapor flow condition. Improper conditions such as foaming, entrainment, weeping/dumping, and flooding can cause significant inefficiencies in the separation. These conditions are often avoided based upon experienced operating design criteria established. Foaming refers to the expansion of liquid due to passage of vapor, or gas. Although

it provides high interfacial liquid-vapor contact, excessive foaming often leads to liquid buildup on trays. In some cases, foaming may be so excessive that the foam mixes with liquid on the tray above. Whether foaming will occur depends primarily on the physical properties of the liquid mixtures, but is sometimes due to tray designs and conditions. Whatever the cause, separation efficiency is always reduced. Entrainment refers to the liquid carried by vapor up to the tray above and is again caused by high vapor flow rates. It is detrimental because tray efficiency is reduced: lower volatile material is carried to a plate holding liquid of higher volatility. It could also contaminate high purity distillate. Excessive entrainment can lead to flooding. Weeping is a phenomenon caused by low vapor flow. The pressure exerted by the vapor is insufficient to hold up the liquid on the tray. Therefore, liquid starts to leak through perforations on the tray. Excessive weeping will lead to dumping. That is the liquid on all trays will crash (dump) through to the base of the column (via a domino effect) and the column will have to be re-started. Weeping is indicated by a sharp pressure drop in the column and reduced separation efficiency. Flooding occurs due to excessive vapor flow, causing liquid to be entrained in the vapor up the column. The increased pressure from excessive vapor also backs up the liquid in the downcomer, causing an increase in liquid holdup on the plate above. Depending on the degree of flooding, the maximum capacity of the column may be severely reduced. Flooding is detected by sharp increases in column differential pressure and significant decrease in separation efficiency.

Many of the above factors that affect column operation are due to vapor flow conditions being either excessive or too low. Vapor flow velocity is dependent on column diameter. Weeping determines the minimum vapor flow required while flooding determines the maximum vapor flow allowed, hence column capacity. Thus, if the column diameter is not sized properly, the column will not perform well. Not only will operational problems occur, the desired separation duties may not be achieved.

The actual number of trays needed for a particular separation duty depends on the efficiency of the plate, and the packings if they are used. Thus, any factors that cause a decrease in tray efficiency will also change the performance of the column. Tray efficiencies are affected by such factors as fouling, wear and tear and corrosion, and the rates at which these occur depends on the properties of the liquids being processed. Thus the proper materials of construction must be selected for tray construction.

A final consideration is weather conditions. Most distillation columns are open to the atmosphere. Although many of the columns are insulated, varying weather conditions can affect column operation. As such, the reboiler must be appropriately sized to ensure that enough vapor can be generated during cold and windy spells and that it can be turned down sufficiently during hot seasons. The same guideline applies to condensors. Other factors to consider include changing operating conditions and throughputs, brought about by changes in upstream conditions and

changes in the demand for the products. These factors, including the associated control system, should be considered at the design stages because once a column is built and installed, nothing much can be done to rectify the situation without incurring additional significant costs.

With the above as an elementary background to the subject of distillation, we will turn our attention to refinery operations and the equipment typically used. Before doing so, a discussion of the properties of hydrocarbons is provided.

GENERAL PROPERTIES OF HYDROCARBONS

Hydrocarbons are compounds containing only hydrogen and carbon atoms. Since a hydrocarbon is a chemical combination of hydrogen and carbons, both of which are non-metals, hydrocarbons are convalently bonded. Hydrogen has only one electron in the outer ring and, therefore, will form only one bond, by donating one electron to the bond. Carbon, on the other hand, occupies a unique position in the Periodic Table, being halfway to stability with its four electrons in the outer ring. None of these electrons are paired, so carbon uses all of them to form covalent bonds. Carbon's unique structure makes it the basis of organic chemistry.

Carbon not only combines covalently with other non-metals, but also with itself. Oxygen also reacts with itself to form O_2, hydrogen reacts with itself to form H_2, nitrogen reacts with itself to form N_2, fluorine reacts with itself to form F_2, and chlorine reacts with itself to form Cl_2. Forming diatomic molecules, however, is the extent of the self-reaction of the elemental gases, while carbon has the ability to combine with itself almost indefinitely. Although the elemental gases form molecules when they combine with themselves, the carbon-to-carbon combination must include another element or elements, generally hydrogen. This combination of carbon with itself (plus hydrogen) forms a larger molecule with every carbon atom that is added to the chain. When the chain is strictly carbon-to-carbon with no branching, the resulting hydrocarbon is referred to as a straight-chain hydrocarbon. Where there are carbon atoms joined to carbon atoms to form side branches off the straight chain, the resulting compound is known as a branched hydrocarbon, or an isomer.

The carbon-to-hydrogen bond is always a single bond. While the resulting bond between carbon and hydrogen is always a single bond, carbon does have the capability to form double and triple bonds between itself and other carbon atoms, and/or any other atom that has the ability to form more than one bond. When a hydrocarbon contains only single bonds between carbon atoms, it is known as a saturated hydrocarbon; when there is at least one double or triple bond between two carbon atoms anywhere in the molecule, it is an unsaturated hydrocarbon. When determining the saturation or unsaturation of a hydrocarbon, only the carbon-to-

carbon bonds are considered, since the carbon-to-hydrogen bond is always single. Hydrocarbons are among the most useful materials to mankind, but are also among the most dangerous in terms of their fire potential.

An analogous series of hydrocarbons, and one of the simplest, are the compounds known as the *alkanes*. In this series, the names of all the compounds end in -ane. The first compound in this series is methane. Methane's molecular formula is CH_4. Methane is a gas and is the principal ingredient in the mixture of gases known as natural gas. The next compound is this series is ethane, whose molecular formula is C_2H_6. It is also a gas present in natural gas, although in a much lower percentage than methane. The difference in the molecular formulas of methane and ethane is one carbon and two hydrogen atoms.

Propane is the next hydrocarbon in this series, and its molecular formula is C_3H_8 which is one carbon and two hydrogen atoms different from ethane. Propane is an easily liquified gas which is used as fuel.

The next hydrocarbon in the series is butane, another rather easily liquified gas used as a fuel. Together, butane and propane are known as the LP (liquified petroleum) gases. Butane's molecular formula is C_4H_{10}, which is CH_2 bigger than propane.

Hence, the series begins with a one-carbon-atom compound, methane, and proceeds to add one carbon atom to the chain for each succeeding compound. Since carbon will form four convalent bonds, it must also add two hydrogen atoms to satisfy those two unpaired electrons and allow carbon to satisfy the octet rule, thus achieving eight electrons in the outer ring. In every hydrocarbon, whether saturated or unsaturated, all atoms must reach stability. There are only two elements involved in a hydrocarbon, hydrogen and carbon; hydrogen must have two electrons in the outer ring, and carbon must have eight electrons in the outer ring. Since the carbon-hydrogen bond is always single, the rest of the bonds must be carbon-carbon, and these bonds must be single, double, or triple, depending on the compound.

Continuing in the alkane series (also called the paraffin series because the first solid hydrocarbon in the series is paraffin, or candle wax), the next compound is pentane. This name is derived from the Greek word penta, for five. As its name implies, it has five carbon atoms, and its molecular formula is C_5H_{12}. From pentane on, the Greek prefix for the numbers five, six, seven, eight, nine, ten, and so on are used to name the alkanes, the Greek prefix corresponding to the number of carbon atoms in the molecule. The first four members of the alkane series do not use the Greek prefix method of naming, simply because their common names are so universally accepted: thus the names methane, ethane, propane, and butane.

The next six alkanes are named pentane, hexane, heptane, octane, nonane, and decane. Their molecular formulas are C_5H_{12}, C_6H_{14}, C_7H_{16}, C_8H_{18}, C_9H_{20} and $C_{10}H_{22}$. The alkanes do not stop at the ten-carbon chain however. Since these first ten represent flammable gases and liquids and most of the derivatives of these

compounds comprise the vast majority of hazardous materials encountered, we have no need to go any further in the series. The general formula for the alkanes is C_nH_{2n+2}. The letter n stands for the number of carbon atoms in the molecule. The number of hydrogen atoms then becomes two more than twice the number of carbon atoms. Since there is more than one analogous series of hydrocarbons, one must remember that each series is unique; the alkanes are defined as the analogous series of saturated hydrocarbons with the general formula C_nH_{2n+2}.

Isomers

Within each analogous series of hydrocarbons there exist isomers of the compounds within that series. An isomer is defined as a compound with the same molecular formula as another compound but with a different structural formula. In other words, if there is a different way in which the carbon atoms can align themselves in the molecule, a different compound with different properties will exist.

Beginning with the fourth alkane, butane, we find we can draw a structural formula of a compound with four atoms and ten hydrogen atoms in two ways; the first is as the normal butane exists and the second is as follows, with the name isobutane (refer to Table 1 for properties).

Butane (C_4H_{10}):

```
        H  H  H  H
        |  |  |  |
     H—C–C–C–C—H
        |  |  |  |
        H  H  H  H
```

Isobutane (C_4H_{10}):

```
         H  H  H
         |  |  |
      H—C–C–C—H
         |  |  |
         H  |  H
         H—C–H
            |
            H
```
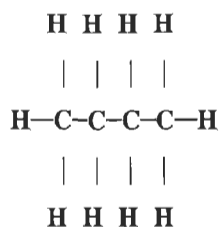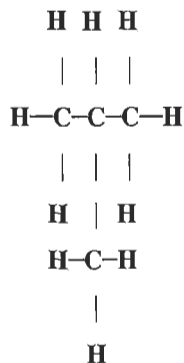
With isobutane, no matter how you count the carbon atoms in the longest chain, you will always end with three. Notice that the structural formula is different - one carbon atom attached to the other carbon atoms - while in butane (also called normal butane), the largest number of carbon atoms another carbon atom can be attached to is two. This fact does make a difference in certain properties of compounds. The molecular formulas of butane and isobutane are the same and, therefore, so are the molecular weights. However, there is a 38-degree difference in melting points, 20-degree difference in boiling points, and the 310-degree difference in ignition temperatures. The structure of the molecule clearly plays part in the properties of the compounds.

With the five-carbon alkane, pentane, there are three ways to draw the structural formula of this compound with five carbon atoms and twelve hydrogen atoms. The isomers of normal pentane are isopentane and neopentane. The structural formulas of these compounds are illustrated in Table 2, while typical properties are given in Table 1.

Table 1. *Typical Properties of Alkanes*[a]

Compound	Formula	Atomic Weight (°F)	Melting Point (°F)	Boiling Point (°F)	Flash Point (°F)	Ignition Temp. (°F)
Methane	CH_4	16	-296.5	-259	gas	999
Ethane	C_2H_6	30	-298	-127	gas	882
Propane	C_3H_8	44	-306	-44	gas	842
Butane	C_4H_{10}	58	-217	31	gas	550
Pentane	C_5H_{12}	72	-201.5	97	< -40	500
Hexane	C_6H_{14}	86	- 139.5	156	-7	437
Heptane	C_7H_{16}	100	-131.1	209	25	399
Octane	C_8H_{18}	114	-70.2	258	56	403
Nonane	C_9H_{20}	128	-64.5	303	88	401
Decane	$C_{10}H_{22}$	142	-21.5	345	115	410
Butane	C_4H_{10}	58	-217	31	gas	550
Isobutane	C_4H_{10}	58	-255	11	gas	860
Pentane	C_5H_{12}	72	-201.5	97	< -40	500
Isopentane	C_5H_{12}	72	-256	82	< -60	788
Neopentane	C_5H_{12}	72	2	49	< -20	842

(a) - values are average literature reported.

Table 2. *Illustrates the Structural Formulas for Isomers of Pentane.*

COMPOUND	MOLECULAR FORMULA	STRUCTURAL FORMULA
Pentane	C_5H_{12}	H H H H H | | | | | H—C–C–C–C–C—H | | | | | H H H H H
Isopentane	C_5H_{12}	H H H H | | | | H—C–C–C–C—H | | | | H H H H H–C–H | H
Neopentane	C_5H_{12}	H | H C H | | | H—C–C–C—H | | | H | H H–C–H | H

Note the three identical molecular formulas and three identical molecular weights, but significantly different melting, boiling, and flash points and different ignition temperatures. These property differences are referred to as the "structural effect", i.e., differences in the properties of compounds exist for materials having the same molecular formulas but different structural arrangements. This particular structure effect is called the branching effect, and the isomers of all the straight-chain hydrocarbons are called branched hydrocarbons. Another structural effect produced is by the length of the chain formed by consecutively attached carbon atoms.

In noting the increasing length of the carbon chain from methane through decane, the difference in each succeeding alkane is that "unit" made up of one carbon atom and two hydrogen atoms; that "unit" is not a chemical compound itself, but it has a molecular weight of fourteen. Therefore, each succeeding alkane in the analogous series weighs fourteen atomic mass units more than the one before it and fourteen less than the one after it. This weight effect is the reason for the increasing melting and boiling points, the increasing flash points, and the decreasing ignition temperatures. The increasing weights of the compounds also account for the changes from the gaseous state of the first four alkanes, to the liquid state of the next thirteen alkanes, and finally to the solid state of the alkanes, starting with the 17-carbon atom alkane, heptadecane.

Note that the larger a molecule (that is, the greater the molecular weight), the greater affinity each molecule will have for each other molecule, therefore, slowing down the molecular movement. The molecules, duly slowed from their frantic movement as gases, become liquids, and, as the molecules continue to get larger, they are further slowed from their still rapid movement as liquids and become solids.

The straight-chain hydrocarbons represent just one group of straight-chain hydrocarbons, the saturated hydrocarbons known as the alkanes. There are other series of hydrocarbons that are unsaturated; one of those is important in the study of hazardous materials. Additionally, the first hydrocarbon in another series is the only hydrocarbon important in that series. Each of these hydrocarbon series are briefly described below.

Alkenes

The series of unsaturated hydrocarbons that contain just one double bond in the structural formula of each of its members is the analogous series known as the alkenes. Notice that the name of the analogous series is similar to the analogous series of saturated hydrocarbons known as the alkanes, but the structural formula is significantly different. Remembering that the definition of a saturated hydrocarbon is a hydrocarbon with nothing but single bonds in the structural formula and that an unsaturated hydrocarbon is a hydrogen-carbon with at least one multiple bond in the structural formula, then we would expect to find a multiple bond in the structural formulas of the alkenes. The names of all the hydrocarbons in this series end in -ene. The corresponding names for this series of hydrocarbons is similar to the alkanes, with the only difference being the above-mentioned ending. Thus, in the alkene series ethane becomes ethene, propane is propene, butane is butene; the five-carbon straight-chain hydrocarbon in the alkene series is pentene, as opposed to pentane in the alkane series, and so on.

Note that these compounds are covalently bonded compounds containing only hydrogen and carbon. The differences in their structural formulas are apparent; the alkanes have only single bonds in their structural formulas, while the alkenes have one (and only one) double bond in their structural formulas. There are different numbers of hydrogen atoms in the two analogous series. This difference is due to the octet rule that carbon must satisfy. Since one pair of carbon atoms shares a double bond, this fact reduces the number of electrons the carbons need (collectively) by two, so there are two fewer hydrogen atoms in the alkene than in the corresponding alkane.

In any hydrocarbon compound, carbon will form four covalent bonds. In saturated hydrocarbons the four bonds will all be single bonds. The definition of an unsaturated hydrocarbon, however, is a hydrocarbon with at least one multiple bond, and the alkenes are an analogous series of unsaturated hydrocarbons containing just one double bond (which is a multiple bond). The double bond must be formed with another carbon atom since hydrogen atoms can form only single bonds and, in a hydrocarbon compounds there are no other elements but hydrogen and carbon. In forming a double bond with another carbon atom and to satisfy the octet rule, the alkene must form fewer bonds with hydrogen, resulting in less hydrogen in the structural formula of each alkene than in the corresponding alkane.

There are two fewer hydrogen atoms in each of the alkenes than in the alkane with the same number of carbon atoms. This is also shown by the general molecular formula of the alkenes, C_nH_{2n}, as opposed to the general molecular formula of the alkanes, which is C_nH_{2n+2}.

Note that there is no one-carbon alkene corresponding to methane, since hydrogen can never form more than one covalent bond, and there is no other carbon atom in the structural formula. Therefore, the first compound in the alkene series is ethene, while the corresponding two-carbon compound in the alkane series, ethane, is the second compound in the series, with methane the first.

Although the naming of the alkenes is the same as the alkanes, with only the ending changed from -ane to -ene, there is a problem with the names of the first three alkenes. The systematic names of hydrocarbons came a long while after the simplest (that is, the shortest chain) of the compounds in each series was known and named. In naming the alkanes, the system of using the Greek names for numbers as prefixes begins with pentane, rather than with methane. That situation occurred because methane, ethane, propane, and butane were known and named long before it was known that there was an almost infinite length to the chain that carbon could form and that a systematic naming procedure would be needed. Before the new system was adopted, the common names for the shortest-chain compounds had become so entrenched that those names survived unchanged. Therefore, not only are the first four compounds in the alkane series named differently from the rest of the series, the corresponding two-, three-, and four-carbon compounds are not generally

known as ethene, propene, and butene. Their common names are ethylene, propylene, and butylene.

As noted earlier, more than one compound may have the same molecular formula (isomers), but a structural formula is unique to one compound. In addition, there are many chemicals which possess more than one chemical name, for the same reason mentioned above. The most common organic chemicals are those that have the shortest carbon chains. This fact is also true of their derivatives. The inclusion of a double bond in the structural formula has a profound effect on the properties of a compound. Table 3 illustrates those differences through the properties of alkenes. The presence of a double bond (and, indeed, a triple bond) between two carbon atoms in a hydrocarbon increases the chemical activity of the compound tremendously over its corresponding saturated hydrocarbon. The smaller the molecule (that is, the shorter the chain), the more pronounced this activity is. A case in point is the unsaturated hydrocarbon ethylene. Disregarding the present the differences in combustion properties between it and ethane, ethylene is so chemically active that, under the proper conditions, instead of burning, polymerization, which, if it is uncontrolled, is a much more violent reaction than combustion. This tendency to polymerize is due to the presence of the double bond. The tendency to polymerize decreases as the molecule gets bigger (the chain is longer). Only the first four or five of the straight-chain hydrocarbons are important in the study of hazardous materials. Few, if any, of the isomers of the alkenes are common.

There are other hydrocarbon compounds that contain multiple bonds, however, discussion here is limited to those compounds containing just one multiple bond in their molecules. This is because the compounds containing just one multiple bond are the most valuable commercially and, therefore, the most common. There is, however, a simple way to recognize when you are dealing with a compound that may contain two double bonds; that is a name in which the Greek prefix "di-" is used. As example would be the compound butadiene. Recognize from the first part of the name ("buta-") that there are four carbon atoms in the chain, and that there is a double bond present (the ending "-ene"), however, just before the -ene ending is the prefix "di-," meaning two. Therefore, recognize that you are dealing with a four-carbon hydrocarbon with two double bonds.

As in the alkanes, it is possible for carbon atoms to align themselves in different orders to form isomers. Not only is it possible for the carbon atoms to form branches which produce isomers, but it is also possible for the double bond to be situated between different carbon atoms in different compounds. This different position of the double bond also results in different structural formulas, which, of course, are isomers. Just as in the alkanes, isomers of the alkenes have different properties. The unsaturated hydrocarbons and their derivatives are more active chemically than the saturated hydrocarbons and their derivatives.

Table 3. *Typical Properties of Alkenes*

Compound	Formula	Molecular Weight	Melting Point (°F)	Boiling Point (°F)	Flash Point (°F)	Ignition Temp. (°F)
Ethylene	C_2H_4	28	-272.2	-155.0	gas	1,009
Propylene	C_3H_6	42	-301.4	-53.9	gas	927
1-Butene	C_4H_8	56	-300.0	21.7	gas	700
2-Butene	C_4H_8	56	-218.2	38.7	gas	615
1-Pentene	C_5H_{10}	70	-265.0	86.0	32	523
2-Pentene	C_5H_{10}	70	-292.0	98.6	32	NA
1-Hexene	C_6H_{12}	84	-219.6	146.4	-15	487
2-Hexene	C_6H_{12}	84	-230.8	154.4	-5	473
1-Heptene	C_7H_{14}	98	-119.2	199.9	28	500
1-Octene	C_8H_{16}	112	-152.3	250.3	70	446

NA = Not Applicable

Alkynes

Another analogous series of unsaturated hydrocarbons that contain just one multiple bond, but, instead of being a double bond, it is a triple bond is the alkynes. The names of all the compounds end in -yne. The only compound in this series that is at all common happens to be an extremely hazardous material. It is a highly unstable (to heat, shock, and pressure), highly flammable gas that is the first compound in the series. This two-carbon unsaturated hydrocarbon with a triple bond between its two carbon atoms is called ethyne, and indeed this is its proper name. It is, however, known by its common name, acetylene.

The -ene ending could be confusing, so one must memorize the fact that acetylene is an alkyne rather than an alkene. Its molecular formula is C_2H_2. The fact that it contains this triple bond makes it extremely active chemically, that is what is meant by its instability to heat, shock, and pressure. It takes energy to start a chemical reaction, and heat, shock, and pressure are forms of energy. The fact that the triple bond contains so much energy tied up in the structure means that it will release this energy, which is the input of some slight amount of external energy. When this input energy strikes the molecule of acetylene, the triple bond breaks, releasing the internal energy of the bonds. This produces either great amounts of heat or an explosion, depending on the way in which the external energy was applied.

There are no other alkynes that are of commercial importance, and so acetylene will be the only member of this series that is considered in fire discussions. There are

other alkynes, however, along with hydrocarbons that might have one double bond and a triple bond present in the molecule.

Straight-Chain Hydrocarbon Nomenclature

The system for naming the straight-chain hydrocarbons is based on an agreed-upon method of retaining the first three or four common names, then using Greek prefixes that indicate the number of carbon atoms in the chain. For isomers, the same system is used, always using the name of the compound that is attached to the chain and the name of the chain.

Recall the first analogous series of hydrocarbons the alkanes, a series of saturated hydrocarbons, all ending in -ane. For these hydrocarbons and other hydrocarbons to react, a place on the hydrocarbon chain must exist for the reaction to take place. Since all the bonds from carbon to hydrogen are already used, an "opening" on one of the carbon atoms must exist for it to be able to react with something else. This "opening" occurs when one of the hydrogen atoms is removed from its bond with a carbon atom, thus causing that carbon to revert back to a condition of instability, with seven electrons in its outer ring, or, as we now state, with one unpaired electron. This one unpaired electron (or half of a covalent bond, or "dangling" bond) wants to react with something, and it will, as soon as another particle which is ready to react is brought near. This chain of carbon atoms (from one carbon to another to another, and so on) with a hydrogen atom missing is a particle that was once a compound, and its name is a *radical*.

Radicals are created by energy being applied to them in a chemical reaction or in a fire. Remember that a hydrocarbon compound with at least one hydrogen atom removed is no longer a compound, but a chemical particle known as a radical. Radicals have names of their own; they are derived from the name of the alkane. When a hydrogen atom is removed from the alkane hydrocarbon, the name is changed from -ane to -yl. Therefore, when a hydrogen is removed from the compound methane, the methyl radical is formed. When a hydrogen atom is removed from the compound ethane, the ethyl radical is formed. In the same manner, the propyl radical comes from propane, the butyl radical comes from butane, and so on. Similarily, isobutane will produce the isobutyl radical, and isopentane will produce the isopentyl radical. A list of hydrocarbons and the radicals produced from them when a hydrogen is removed is shown is Table 4. Note that there are only a few radicals from compounds other than the alkanes which are important. Radicals are referred to as hydrocarbon "backbones". As an example, isobutane is more properly named methyl propane. Another isomer with a different proper name is isopentane, more properly called methyl butane. Neopentane is also named 2,2-dimethyl propane.

Table 4. *A Listing of Common Radicals*

Methane	CH_4	Methyl	$-CH_3$
Ethane	C_2H_6	Ethyl	$-C_2H_5$
Propane	C_3H_8	n-Propyl	$-C_3H_7$
		Isopropyl	$-C_3H_7$
Butane	C_4H_{10}	n-Butyl	$-C_4H_9$
Isobutane	C_4H_{10}	Isobutyl	$-C_4H_9$
		sec-Butyl	$-C_4H_9$
		tert-Butyl	$-C_4H_9$
Ethylene	C_2H_4	Vinyl	$-C_2H_3$
Benzene	C_6H_6	Phenyl	$-C_6H_5$

The following is a list of rules for proper nomenclature of the isomers and their derivatives.

1. Find the longest continuous chain and name it as if it were an alkane.

2. Name the side branches in the same manner.

3. Identify the number of the carbon atom on the longest chain to which the branch is attached by counting from the end of the chain nearest to the branch.

4. If there could is any confusion as to which carbon atom is meant, put the number in front of the name of the compound, followed by a dash.

5. If there is more than one branch, use the numbers to identify the carbon atom to which they are attached.

6. If the branches are identical, use the prefixes di- for two, tri- for three, tetra- for four, and so on.

In this manner, the four isomers of hexane are named 2-methyl pentane, 3-methyl pentane, 2,2,-dimethyl butane, and 2,3-dimethyl butane.

Aromatic Hydrocarbons

The above discussions have concentrated on hydrocarbons, both saturated and unsaturated, with the unsaturated hydrocarbons containing only one multiple bond. The unsaturated hydrocarbons are the alkenes with one double bond and the alkynes

with one triple bond. There are other straight-chain hydrocarbons that are unsaturated containing more than one multiple bond, some with more than one double bond, and some with a mixture of double bonds and triple bonds. The combinations and permutations are endless, but there are only a few of the highly unstable materials.

From a commercial standpoint, there is a large body of hydrocarbons that is very important. These hydrocarbons are different in that they are not straight-chain hydrocarbons but have a structural formula that can only be called cyclical. The most common and most important hydrocarbon in this group is benzene. It is the first and simplest of the six-carbon cyclical hydrocarbons referred to as aromatic hydrocarbons.

Benzene's molecular formula is C_6H_6, but it does not behave like hexane, hexene, or any of their isomers. One would expect it to be similar to these other six-carbon hydrocarbons in its properties. Table 5 provides a comparison between benzene, hexane and 1-hexene. The table shows that there are major differences between benzene and the straight-chain hydrocarbons of the same carbon content. Hexene's ignition temperature is very near to hexane's. The flash point difference is not great, however, there are significant differences in melting points. The explanation for these differences is structure; which in the case of benzene is a cyclical form with alternating double bonds.

The alternating double bonds are illustrated in Figure 17A. Initially, it was believed that the alternating double bonds impart very different properties to benzene, however, and the fact is that they do not. The only possible way for the benzene molecule to exist is illustrated in Figure 17B, in which a circle is drawn within the hexagonal structural to show that the electrons that should form a series of alternating double bonds are really spread among all six carbon atoms. It is the only structure possible that would explain the unique properties of benzene. This structural formula suggests resonance; that is, the possibility that the electrons represented by the circle are alternating back and forth between and among the six carbon atoms.

Table 5. *Compares Between Benzene and of Straight-Chain Hydrocarbons.*

Compound	Formula	Melting Point (°F)	Boiling Point (°F)	Flash Point (°F)	Ignition Temp. (°F)	Molecular weight
Hexane	C_6H_{14}	-139.5	156.0	-7	500	86
1-Hexene	C_6H_{12}	-219.6	146.4	<-20	487	84
Benzene	C_6H_6	41.9	176.2	12	1,044	78
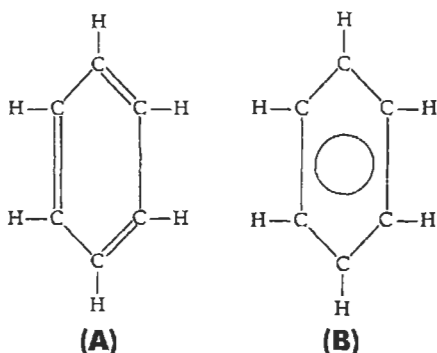
Figure 17. *The structure of benzene: (A) conventional illustration of double bonds, (B) illustration implying resonance.*
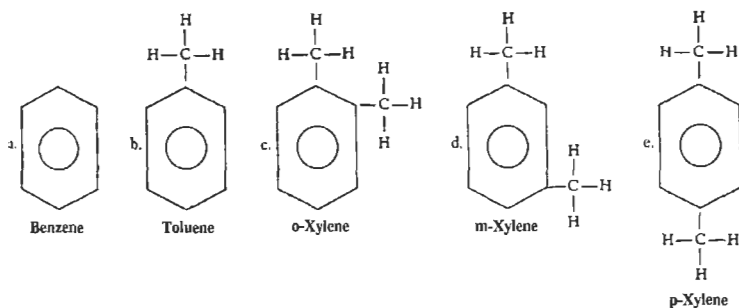
Figure 18. *The structures of benzene and some of its common derivatives.*

This particular hexagonal structure is found throughout nature in many forms, almost always in a more complicated way, usually connected to many other "benzene rings" to form many exotic compounds. Of importance to the immediate discussions are benzene and a few of its derivatives. Benzene's derivatives include toluene and xylene, whose structural formulas are illustrated in Figure 18 along with that of benzene. Some typical properties are given in Table 6, which illustrates the differences caused by molecular weight and structural formulas. There are other cyclical hydrocarbons, but they do not have the structural formulas of the aromatics, unless they are benzene-based. These cyclical hydrocarbons may have three, four, five, or seven carbons in the cyclical structure, in addition to the six-carbon ring of the aromatics. None of them has the stability or the chemical properties of the aromatics.

Table 6. *Comparison of Benzene and Some of its Derivatives,*

Compound	Formula	Melting Point (°F)	Boiling Point (°F)	Flash Point (°F)	Ignition Temperature (°F)	Molecular Weight
Benzene	C_6H_6	41.9	176.2	12	1,044	78
Toluene	C_7H_8	-138.1	231.3	40	997	92
o-xylene	C_8H_{10}	-13.0	291.2	90	867	106
m-xylene	C_8H_{10}	-53.3	281.9	81	982	106
p-xylene	C_8H_{10}	-55.8	281.3	81	984	106

The aromatic hydrocarbons are used mainly as solvents and as feedstock chemicals for chemical processes that produce other valuable chemicals. With regard to cyclical hydrocarbons, the aromatic hydrocarbons are the only compounds discussed. These compounds all have the six-carbon benzene ring as a base, but there are also three-, four-, five-, and seven-carbon rings. These materials will be considered as we examine their occurrence as hazardous materials. After the alkanes, the aromatics are the next most common chemicals shipped and used in commerce. The short-chain olefins (alkenes) such as ethylene and propylene may be shipped in larger quantities because of their use as monomers, but for sheer numbers of different compounds, the aromatics will surpass even the alkanes in number, although not in volume.

Hydrocarbon Derivatives

A hydrocarbon derivative is a compound with a hydrocarbon backbone and a functional group attached to it chemically. A hydrocarbon backbone is defined as a molecular fragment that began as a hydrocarbon compound and has had at least one hydrogen atom removed from the molecule. Such a fragment is also known as a radical. A functional group is defined as an atom or a group of atoms, bound together, which impart specific chemical properties to a molecule; also referred to as radicals. A hydrocarbon derivative then is essentially a compound made up of two specific parts; the first part comes from a hydrocarbon, and the second may have many different origins (which includes coming from a hydrocarbon), depending on the chemical makeup of the functional group. The hydrocarbon backbone may come from an alkane, an alkene, an alkyne (indeed, any saturated or unsaturated hydrocarbon), or from an aromatic hydrocarbon or other cyclical hydrocarbon. Any hydrocarbon compound may form the hydrocarbon backbone

portion of the hydrocarbon derivative, as long as it has been converted to a radical, by removal of one or more hydrogens, in preparation for the reaction. The functional group may have many origins, with chemists using as reactants any chemical compound that will produce the desired functional group. The functional groups include the halogens (fluorine, chlorine, bromine, and iodine), the hydroxyl radical, the carbonyl group, oxygen, the carboxyl group, the peroxide radical, the amine radical, and even other hydrocarbon radicals. When these functional groups are chemically attached to hydrocarbon backbones, they form compounds called hydrocarbon derivatives, and each functional group imparts a separate set of chemical and physical properties to the molecule formed by this chemical attachment.

Just as the alkanes and alkenes had general formulas, the carbon derivatives all have general formulas. The hydrocarbon backbone provides a portion of the general formula, and the functional group provides the other part. In each case, the hydrocarbon derivative is represented by the formula R-, and the hydrocarbon backbone has its own specific formula. The term "substituted hydrocarbon" is another name for hydrocarbon derivative, because the functional group is substituted for one or more hydrogen atoms in the chemical reaction.

Halogenated Hydrocarbons

A halogenated hydrocarbon is defined as a derivative of a hydrocarbon in which a hydrogen atom is replaced by a halogen atom. Since all of the halogens react similarly, and the number of hydrocarbons (including all saturated hydrocarbons, unsaturated hydrocarbons, aromatic hydrocarbons, other cyclical hydrocarbons, and all the isomers of these hydrocarbons) is large, the number of halogenated hydrocarbons can also be very large. The most common hydrocarbon derivatives are those of the first four alkanes and the first three alkenes (and, of course, the isomers of these hydrocarbons). There are some aromatic hydrocarbon derivatives, but, again, they are of the simplest structure. Whatever the hydrocarbon backbone is, it is represented in the general formula by its formula, which is R-. Therefore, the halogenated hydrocarbons will have formulas such as R-F, R-Cl, R-Br, and R-I for the respective substitution of fluorine, chlorine, bromine, and iodine on to the hydrocarbon backbone. As a rule, the general formula can be written R-X, with the R as the hydrocarbon backbone, the X standing for the halide (any of the halogens), and the ''-'' the covalent bond between the hydrocarbon backbone and the halogen. R-X is read as "alkyl halide".

Radicals of the alkanes are referred to as alkyl radicals. There are two other important radicals; they are the vinyl radical, which is produced when a hydrogen atom is removed from ethylene, and the phenyl radical, which results when a

hydrogen atom is removed from benzene. The term halogenated means that a halogen atom has been substituted for a hydrogen atom in a hydrocarbon molecule. The most common halogenated hydrocarbons are the chlorinated hydrocarbons. The simplest chlorinated hydrocarbon is methyl chloride, whose molecular formula is CH_3Cl. The structural formula for methyl chloride shows that one chlorine atom is substituted for one hydrogen atom. Methyl chloride has many uses, such as a herbicide, as a topical anesthetic, extractant, and low-temperature solvent, and as a catalyst carrier in low-temperature polymerization. It is a colorless gas that is easily liquified and is flammable; it is also toxic in high concentrations. Methyl chloride is the common name for this compound, while its proper name is chloromethane. Proper names are determined by the longest carbon chain in the molecule, and the corresponding hydrocarbon's name is used as the last name of the compound. Any substituted groups are named first, and a number is used to designate the carbon atom that the functional group is attached to, if applicable.

It is possible to substitute more than one chlorine atom for a hydrogen atom on a hydrocarbon molecule; such substitution is done only when the resulting compound is commercially valuable or is valuable in another chemical process. An example is methylene chloride (the common name for dichloromethane), which is made by substituting two chlorine atoms for two hydrogen atoms on the methane molecule. Its molecular formula is CH_2Cl_2. Methylene chloride is a colorless, volatile liquid with a sharp, ether-like odor. It is listed as a non-flammable liquid, but it will ignite at $1,224°F$; it is narcotic at high concentrations. It is most commonly used as a stripper of paints and other finishes. It is also a good degreaser and solvent extractor and is used in some plastics processing applications.

Substituting a third chlorine on the methane molecule results in the compound whose proper name is trichloromethane (tri- for three; chloro- for chlorine; and methane, the hydrocarbon's name for the one-carbon chain). It is more commonly known as chloroform. Its molecular formula is $CHCl_3$. Chloroform is a heavy, colorless, volatile liquid with a sweet taste and characteristic odor. It is classified as non-flammable, but it will burn if exposed to high temperatures for long periods of time. It is narcotic by inhalation and toxic in high concentrations. It is an insecticide and a fumigant and is very useful in the manufacture of refrigerants. The total chlorination of methane results in a compound whose proper name is tetrachloromethane (tetra- for four), but its common name is carbon tetrachloride (or carbon tet). This is a fire-extinguishing agent that is no longer used since it has been classified as a carcinogen. It is still present, though, and its uses include refrigerants, metal degreasing, and chlorination of organic compounds. Its molecular formula is CCl_4. It is possible to form analogues of methyl chloride, (methyl fluoride, methyl bromide, methyl iodide), methylene chloride (substitute fluoride, bromide, and iodide in this name also), chloroform (fluoroform, bromoform, and iodoform), and carbon tetrachloride (tetrafluoride, tetrabromide, and tetraiodide). Each of these halogenated hydrocarbons has commercial value.

What was true for one hydrocarbon compound is true for most hydrocarbon compounds, particularly straight-chain hydrocarbons; that is, you may substitute a functional group at each of the bonds where a hydrogen atom is now connected to the carbon atom. Where four hydrogen atoms exist in methane, there are six hydrogen atoms in ethane; you recall that the difference in make-up from one compound to the next in an analogous series is the "unit" made up of one carbon and two hydrogens. Therefore, it is possible to substitute six functional groups on to the ethane molecule. You should also be aware that the functional groups that would be substituted for the hydrogens need not be the same, that is, you may substitute chlorine at one bond, fluorine at another, the hydroxyl radical at a third, an amine radical at a fourth, and so on. Substituting one chlorine atom for a hydrogen atom in ethane produces ethyl chloride, a colorless, easily liquifiable gas with an ether-like odor and a burning taste, which is highly flammable and moderately toxic in high concentrations. It is used to make tetraethyl lead and other organic chemicals. Ethyl chloride is an excellent solvent and analytical reagent, as well as an anesthetic. Its molecular formula is C_2H_5Cl. Although we are using chlorine as the functional group, it may be any of the other halogens. In addition, we are giving the common names, while the proper names may be used on the labels and shipping papers. Ethyl chloride's proper name is chloroethane.

Substituting another chlorine produces ethylene dichloride (proper name 1,2-dichloroethane). In this case, an isomer is possible, which would be the chlorinated hydrocarbon where both chlorines attached themselves to the same carbon atom, whereby 1,1-dichloroethane is formed. These compounds have slightly different properties and different demands in the marketplace. As further chlorination of ethane occurs, we would have to use the proper name to designate which compound is being made. One of the analogues of ethylene dichloride is ethylene dibromide, a toxic material that is most efficient and popular as a grain fumigant, but it is known to be a carcinogen in test animals. There are many uses for the halogenated hydrocarbons. Many of them are flammable; most are combustible. Some halogenated hydrocarbons are classified as neither, and a few are excellent fire-extinguishing agents (the Halons ®), but they will all decompose into smaller, more harmful molecular fragments when exposed to high temperatures for long periods of time.

Alcohols

The compounds formed when a hydroxyl group (-OH) is substituted for a hydrogen are called alcohols. They have the general formula R-OH. The hydroxyl radical looks exactly like the hydroxide ion, but it is not an ion. Where the hydroxide ion fits the definition of a complex ion - a chemical combination of two or more atoms that have collectively lost or (as in this case) gained one or more electrons - the

hydroxide radical is a molecular fragment produced by separating the -OH from another compound, and it has no electrical charge. It does have an unpaired electron waiting to pair up with another particle having its own unpaired electron. The alcohols, as a group, are flammable liquids in the short-chain range, combustible liquids as the chain grows longer, and finally solids that will burn if exposed to high temperatures, as the chain continues to become longer. As in the case of the halogenated hydrocarbons, the most useful alcohol compounds are of the short-carbon-chain variety. Just as in the case of the halogenated hydrocarbons, the simplest alcohol is made from the simplest hydrocarbon, methane. Its name is methyl alcohol and its molecular formula is CH_3OH.

Nature produces a tremendous amount of methyl alcohol, simply by the fermentation of wood, grass, and other materials made to some degree of cellulose. In fact, methyl alcohol is known as wood alcohol, along with names such as wood spirits and methanol (its proper name; the proper names of all alcohols end in -ol). Methyl alcohol is a colorless liquid with a characteristic alcohol odor. It has a flash point of 54°F, and is highly toxic. It has too many commercial uses to list here, but among them are as a denaturant for ethyl alcohol (the addition of the toxic chemical methyl alcohol to ethyl alcohol in order to form denatured alcohol), antifreezes, gasoline additives, and solvents. No further substitution of hydroxyl radicals is performed on methyl alcohol.

The most widely known alcohol is ethyl alcohol, simply because it is the alcohol in alcoholic drinks. It is also known as grain alcohol, or by its proper name, ethanol. Ethyl alcohol is a colorless, volatile liquid with a characteristic odor and a pungent taste. It has a flash point of 55°F, is classified as a depressant drug, and is toxic when ingested in large quantities. Its molecular formula is C_2H_5OH. In addition to its presence in alcoholic beverages, ethyl alcohol has many industrial and medical uses, such as a solvent in many manufacturing processes, as antifreeze, antiseptics, and cosmetics.

The substitution of one hydroxyl radical for a hydrogen atom in propane produces propyl alcohol, or propanol, which has several uses. Its molecular formula is C_3H_7OH. Propyl alcohol has a flash point of 77°F and, like all the alcohols, burns with a pale blue flame. More commonly known is the isomer of propyl alcohol, isopropyl alcohol. Since it is an isomer, it has the same molecular formula as propyl alcohol but a different structural formula. Isopropyl alcohol has a flash point of 53°F. Its ignition temperature is 850°F, while propyl alcohol's ignition temperature is 700°F, another effect of the different structure. Isopropyl alcohol, or 2-propanol (its proper name) is used in the manufacture of many different chemicals, but is best known as rubbing alcohol.

The above-mentioned alcohols are by far the most common. Butyl alcohol is not as commonly used as the first four in the series, but it is used. Secondary butyl alcohol and tertiary butyl alcohol, so named because of the type of carbon atom in the

molecule to which the hydroxyl radical is attached, must be mentioned because they are flammable liquids, while isobutyl alcohol has a flash point of 100 °F. All of the alcohols of the first four carbon atoms in the alkanes, therefore, are extremely hazardous because of their combustion characteristics.

Whenever a hydrocarbon backbone has two hydroxyl radicals attached to it, it becomes a special type of alcohol known as a glycol. The simplest of the glycols, and the most important, is ethylene glycol, whose molecular formula $C_2H_4(OH)_2$. The molecular formula can also be written CH_2OHCH_2OH and may be printed as such on some labels. Ethylene glycol is a colorless, thick liquid with a sweet taste, is toxic by ingestion and by inhalation, and among its many uses is a permanent antifreeze and coolant for automobiles. It is a combustible liquid with a flash point of 240°F.

The only other glycol that is fairly common is propylene glycol which has a molecular formula of $C_3H_6(OH)_2$. It is a combustible liquid with a flash point of 210°F, and its major use is in organic synthesis, particularly of polyester resins and cellophane.

The last group of substituted hydrocarbons produced by adding hydroxyl radicals to the hydrocarbon backbone are the compounds made when three hydroxyl radicals are substituted; these are known as glycerols. The name of the simplest of this type of compound is just glycerol. Its molecular formula is $C_3H_5(OH)_3$. Glycerol is a colorless, thick, syrupy liquid with a sweet taste, and has a flash point of 320°F, and is used to make such diverse products as candy and explosives, plus many more. Other glycerols are made, but most of them are not classified as hazardous materials.

Ethers

The ethers are a group of compounds with the general formula R-O-R'. The R, of course, stands for any hydrocarbon backbone, and the R' also stands for any hydrocarbon backbone, but the designation R' is used to indicate that the second hydrocarbon backbone may be different from the first. In other words, both the hydrocarbon backbones in the formula may be the same, but the "'" is used to indicate that it may also be different. R-O-R as the general formula for the ethers is also correct. The fact that there are two hydrocarbon backbones on either side of an oxygen atom means that there will be two hydrocarbon names used.

The simplest of the ethers would be ether that has the simplest hydrocarbon backbones attached; those backbones are the radicals of the simplest hydrocarbon, methane. Therefore, the simplest of the ethers is dimethyl ether, whose formula is CH_3OCH_3. Dimethyl is used because there are two methyl radicals, and "di-" is the prefix for two. This compound could also be called methyl methyl ether, or just

plain methyl ether, but it is better known as dimethyl ether. It is an easily liquified gas that is extremely flammable, has a relatively low ignition temperature of 66°F, and is used as a solvent, a refrigerant, a propellant for sprays, and a polymerization stabilizer.

The next simplest ether is the ether with the simplest alkane as one of the hydrocarbon backbones and the next alkane, which is methyl ethyl ether. Its molecular formula is $CH_3OC_2H_5$. It is a colorless gas with the characteristic ether odor. It has a flash point of 31°F, and an ignition temperature of only 374°F. This property, of course, makes it an extreme fire and explosion hazard.

The next simplest ether is actually the one most commonly referredto as "ether". It is diethyl ether, whose molecular formula is $C_2H_5OC_2H_5$, sometimes written as $(C_2H_5)_2O$. This ether is the compound that was widely used as an anesthetic in many hospitals. One of the hazards of all ethers, and particularly diethyl ether because of its widespread use, is that once ethers have been exposed to air, they possess the unique capability of adding an oxygen atom to their structure and converting to a dangerously unstable and explosive organic peroxide. The peroxide-forming hazard aside, diethyl etherhas a flash point of -56°F and ignition temperature of 356 °F; it is a colorless, volatile liquid with the characteristic ether odor. In addition to its use as an anesthetic, it is useful in the synthesis of many other chemicals, but it is an extremely hazardous material.

Another important ether is vinyl ether, a colorless liquid with the characteristic ether odor. Its molecular formula is $C_2H_3OC_2H_3$. Vinyl ether has a flash point of -22°F and an ignition temperature of 680°F. It is highly toxic by inhalation and is used in medicine and in the polymerization of certain plastics.

Ketones

The ketones are a group of compounds with the general formula R-C-R'. The -C-functional group is known as the carbonyl group or carbonyl radical; it appears in many different classes of hydrocarbon derivatives. There are only a few important ketones, and they are all extremely hazardous.

The first is the simplest, again with two methyl radicals, one on either side of the carbonyl group. Its molecular formula is CH_3COCH_3. Its proper name is propanone (propa- because of the relationship to the three-carbon alkane, propane, and -one because it is a ketone); it could logically be called dimethyl ketone, but it is universally known by its common name, acetone. Acetone is a colorless, volatile liquid with a sweet odor, has a flash point of 15°F and an ignition temperature of 1,000° F, is narcotic in high concentrations, and could be fatal by inhalation or ingestion. It is widely used in manufacturing many chemicals and is extremely popular as a solvent.

The next most common ketone is methyl ethyl ketone, commonly referred to as MEK. Its molecular formula is $CH_3COC_2H_5$. MEK has a flash point of 24°F and an ignition temperature of 960°F. It is a colorless liquid with a characteristic ketone odor. It is as widely used as acetone and is almost as hazardous.

Aldehydes

The aldehydes are a group of compounds with the general formula R-CHO. The aldehyde functional group is always written -CHO, even though this does not represent the aldehyde's structural formula. It is written in this way so that the aldehydes will not be confused with R-OH, the general formula of the alcohols. The simplest of the aldehydes is formaldehyde, whose molecular formula is HCHO. The second hydrocarbon backbone of the ketone is replaced by a hydrogen atom. Formaldehyde is a gas that is extremely soluble in water; it is often sold commercially as a 50 percent solution of the gas in water. The gas itself is flammable, has an ignition temperature of 806°F and a strong, pungent odor, and is toxic by inhalation. Inhalation at low concentrations over long periods of time has produced illness in many people. Beside its use as an embalming fluid, formaldehyde is used in the production of many plastics and in the production of numerous other chemicals. The next aldehyde is acetaldehyde, a colorless liquid with a pungent taste and a fruity odor. Its molecular formula is CH_3CHO. It has a flash point of -40°F, an ignition temperature of 340°F, and is toxic by inhalation. Acetaldehyde is used in the manufacture of many other chemicals. Other important aldehydes are propionaldehyde, butyraldehyde, and acrolein.

Peroxides

The peroxides are a group of compounds with the general formula R-O-O-R'. All peroxides are hazardous materials, but the organic peroxides may be the most hazardous of all.

Esters

The esters are a group of compounds with the general formula R-C-O-O-R'. They are not generally classified as hazardous materials, except for the acrylates, which are monomers and highly flammable. Few of the rest of the class are flammable. There are some esters that are hazardous.

Amines

The amines are a group of compounds with the general formula $R-NH_2$, and all the common amines are hazardous. As a class the amines pose more than one hazard, being flammable, toxic, and, in some cases, corrosive. The amines are an analogous series of compounds and follow the naming pattern of the alkyl halides and the alcohols; that is, the simplest amine is methyl amine, with the molecular formula of CH_3NH_2. Methyl amine is a colorless gas with an ammonia-like odor and an ignition temperature of 806°F. It is a tissue irritant and toxic, and it is used as an intermediate in the manufacture of many chemicals. Ethyl amine is next in the series, followed by propyl amine, isopropyl amine, butyl amine and its isomers, and so on.

REFINERY OPERATIONS

Overview of Refinery Operations

Refining breaks crude oil down into its various components, which are then selectively reconfigured into new products. The complexity of operations varies from one refinery to the next. In general, the more sophisticated a refinery, the better its ability to upgrade crude oil into high-value products. All refineries perform three basic steps: separation, conversion and treatment. Modern separation involves piping oil through hot furnaces. The resulting liquids and vapors are discharged into distillation towers. Inside the towers, the liquids and vapors separate into components or fractions according to weight and boiling point. The lightest fractions, including gasoline and liquid petroleum gas (LPG), vaporize and rise to the top of the tower, where they condense back to liquids. Medium weight liquids, including kerosene and diesel oil distillates, stay in the middle. Heavier liquids, called gas oils, separate lower down, while the heaviest fractions with the highest boiling points settle at the bottom. These tarlike fractions, called residuum, are literally the "bottom of the barrel." The fractions now are ready for piping to the next station or plant within the refinery. Some components require relatively little additional processing to become asphalt base or jet fuel. However, most molecules that are destined to become high-value products require much more processing.

It is the ultimate objective of a refinery to transform the fractions from the distillation towers into streams (intermediate components) that eventually become finished products. This also is where a refinery makes money, because only through conversion can most low-value fractions become gasoline. The most widely used conversion method is called cracking because it uses heat and pressure to "crack"

heavy hydrocarbon molecules into lighter ones. A cracking unit consists of one or more tall, thick-walled, bullet-shaped reactors and a network of furnaces, heat exchangers and other vessels. Fluid catalytic cracking, or "cat cracking," is the basic gasoline making process. Using intense heat (about 1,000 °F); low pressure and a powdered catalyst, the cat cracker can convert most relatively heavy fractions into smaller gasoline molecules. Hydrocracking applies the same principles but uses a different catalyst, slightly lower temperatures, much greater pressure and hydrogen to obtain chemical reactions. Although not all refineries employ hydrocracking, some companies use this technology to cost-effectively convert medium to heavyweight gas oils into high-value streams.

Some refineries also have cokers, which use heat and moderate pressure to turn residuum into lighter products and a hard, coal-like substance that is used as an industrial fuel. Cokers are among the more peculiar- looking refinery structures. They resemble a series of giant drums with metal derricks on top. Cracking and coking are not the only forms of conversion. Other refinery processes, instead of splitting molecules, rearrange them to add value. Alkylation, for example, makes gasoline components by combining some of the gaseous byproducts of cracking. The process, which essentially is cracking in reverse, takes place in a series of large, horizontal vessels and tall, skinny towers that loom above other refinery structures. Reforming uses heat, moderate pressure and catalysts to turn naphtha, a light, relatively low-value fraction, into high-octane gasoline components.

A major portion of refining involves blending, purifying, fine-tuning and otherwise improving products to meet customer requirements. To make gasoline, refinery technicians carefully combine a variety of streams from the processing units. Among the variables that determine the blend are octane level, vapor pressure ratings and special considerations, such as whether the gasoline will be used at high altitudes. Technicians also add performance additives, and dyes that distinguish the various grades of fuel. By the time a gallon of gasoline is pumped into a car's tank, it contains more than 200 hydrocarbons and additives.

The Role of Fractional Distillation

Raw petroleum (crude oil) is extracted from underground around the globe in a variety of ways and refined for tens of thousands of applications in our everyday lives. Crude oil is refined into usable petroleum products through several unique processes. Fractional distillation is the process used to efficiently extract or "distill" products that are a mixture of chemicals such as gasoline, diesel fuel, and kerosene or may selectively extract pure chemical compounds or petrochemicals such as

butane, benzene, toluene, trichloroethylene, and thousands of others. The fractional distillation process converts crude oil into petroleum products by separating the crude oil into its constituent components through evaporation and condensation. The process of distillation has been known for centuries, as far back as the time of Aristotle. In Meteorologica, he stated, "salt water, when it turns into vapor becomes sweet and the vapor does not form salt water again when it condenses." By the middle ages, monks, doctors, and others had refined the distillation process for use in the creation of healing brews and herbal extracts, starting the distilled alcoholic beverage industry in the process since the healing brews had an alcohol base. Today, the distillation process is used to produce perfume, alcoholic beverages, cooking oils, and, of course, petrochemicals.

The evaporation of salt water to produce sea salt and water from water vapor is simple distillation, in that there is a single evaporation/condensation stage. Simple distillation can be used to extract multiple components of crude oil. The oil could be placed within a container that, when heated to a specific temperature, would evaporate compounds in the crude based on the specific temperature applied to the container. The evaporates would then be routed to a condenser to be returned to liquid form. The temperature could be gradually increased over time to extract less volatile compounds in the crude mix. While this method is basically straightforward, it is labor and energy intensive. The fractional distillation process is more efficient. This process capitalizes on the relative volatility of the chemical compounds in crude oil. A chemical that is highly volatile has a low evaporation point and a chemical that is of lower volatility has a high condensation point. Thus, it is possible to separate the constituent chemicals from the crude oil according to their volatility or condensation temperature. In the fractional distillation process this is accomplished by controlling the temperature of the crude oil mixture over space, a temperature gradient that is warmest closest to the source of the vapor and cooler farther away. By gradually decreasing the temperature along the gradient, the components with the lowest volatility condense at the highest temperature, followed by more volatile compounds at lower temperatures. By using the fractional distillation method, individual compounds or mixtures can be distilled from the crude. Also, the fractional distillation process is more labor and energy efficient because additional crude can be added at any time and multiple compounds can be extracted using one energy source.

Fractional distillation is practically accomplished in a fractionating tower designed specifically for rectifying petroleum products from crude oil by creating a temperature gradient along the height of the tower or stack. In the stack, crude oil is pumped into a boiler and heated. Cascading action increases the surface area of the liquid to aid in rapid evaporation. Condensate trays or plates extend up the height of the tower along the temperature gradient. Highly volatile chemicals condense farthest up the stack and are extracted from the tower via a network of pipelines. The less volatile chemicals condense lower in the stack at higher temperatures. By putting together a system of multiple fractionating towers

operating at different temperatures, it is possible to extract individual compounds from the crude oil mixture. For example, in the first stage, crude oil may be added to the main stack and heated. At a certain height in the stack, a plate is present that gasoline condenses on. The gasoline condensate could then be piped into two pathways: 1) raw gasoline to another refinery to be modified by additives for use in internal combustion engines and 2) raw gasoline into another stack. In the second pathway, the second stack could be operated at a temperature and with specific plates that could rectify the individual chemical compound constituents of the gasoline, such as benzene, ethylbenzene, toluene, and xylenes. This process can be used for any of the general fractions of crude oil to further rectify the constituent compounds. Crude oil can be generally grouped into fractions as follows (in order from most volatile to least volatile):

Natural Gas: The lightest, most volatile petroleum fractions, such as propane, pentane, etc.

Gasolines: Higher distillates used for automotive fuel.

Middle Distillates: Lower volatility distillates including diesel fuel, kerosene, jet fuels, and lighter fuel oils.

Heavier Fuel Oils and Lubricating Oils: Low volatility distillates, e.g., motor oil.

Asphalts and Tars: Lowest volatility and generally a smelly, sticky mess filled with impurities.

Historical Perspectives

A typical large refinery costs billions of dollars to build and millions more to maintain and upgrade. It runs around the clock 365 days a year, employs between 1,000 and 2,000 people and occupies as much land as several hundred football fields. Modern refinery operations had surprisingly humble origins. In 1876, pioneers used wagons and mules to haul primitive stills to a spot near Pico Canyon, the site of California's first producing oil wells. The stills, each about the size of a garage, were used to heat oil at the rate of 25 to 40 barrels a day. This "oil boiling" produced kerosene, lubricants, waxes and gasoline--a clear, lightweight liquid that generally was discarded as a useless byproduct in those days. Gasoline's lowly status rose quickly after 1892, when Charles Duryea built the first U.S. gas-powered automobile.

In the early years of the petroleum industry, distillers discovered the benefits of thermal cracking to produce benzene by forcing their stills to the limits. One of the most successful early catalytic cracking processes was the Houdry process, of which the first commercial unit was placed into operation in 1936. This development significantly increased gasoline yields from crude. The early Houdry process employed a silica alumina catalyst, which at high temperature cracked gas oils to gasoline. The principal drawback with this process was the regeneration of the

catalyst. A byproduct of the cracking reaction is coke, which deposits onto the catalyst and blinds or deactivates the active catalytic sites. Coke can be burned off in air; however, a considerable amount of heat is liberated, which if not removed, can melt the catalyst.

Catalytic cracking in the Houdry process was originally performed in fixed beds. Temperature control in these reactors was achieved using a molten salt heat removal scheme as the catalyst was regenerated. The heat of reaction and some of the required feed preheat were supplied by circulating the molten salt through vertical tubes distributed through the reactor bed. A typical reaction cycle for an individual reactor was about ten minutes. At the end of this cycle, feed was automatically switched to another reactor that had been regenerated. The spent reactor was steam purged for several minutes and then isolated by an automatic cycle timer. Regeneration air was then introduced and the carbon was burned off at a rate at which the bed temperature could be controlled by the recirculating of the molten salt stream. The regenerated bed had to be purged of oxygen before being returned into cracking service. A typical operation consisted of three to six reactors. The main difficulty with the fixed-bed catalytic cracker was that equilibrium was never achieved. The gas oil conversion (i.e., the amount of feed converted to lighter components) was initially high at the start of the reaction and gradually diminished as carbon deposited onto the catalyst until regeneration was needed. To minimize this effect multiple parallel reactors were employed; however, true steady-state operation was never actually achieved. Reaction-bed temperatures during reaction and regeneration cycles fluctuated considerably.

The next major development in gasoline production was a moving bed operation (process flow sheets of two early units are shown in Figure 19). In the moving bed processes, the hot salt heat transfer and cycle time systems were eliminated. The catalyst was transferred to the top of the unit and flowed by gravity down through the vessel. These systems were first commercialized around 1943. The catalyst was pelletized into about one eighth-inch, diameter beads and allowed to flow by gravity from the top of the vessel, down through a seal zone to the pressurized reactor vessel. The catalyst then flowed down through another sealing section and a countercurrent stripping zone. From there, catalyst flowed to the regenerator or kiln, which operated close to atmospheric pressure. The Socony Vacuum Oil Co.'s unit (Figure 19 A) injected the regeneration air near the center of the regenerator bed. The gas flowed upward and then downward. The upflowing gas combusted about 60% of the coke, and heated the downward flowing catalyst. The downward flowing gas completed the combustion process. The principal difference between the Socony Vacuum process (Figure 19 A) and the modified Houdry process (Figure 19 B) was that the latter consisted of a single vessel with reaction and stripping zones separated by intermediate vessel heads. Also, flue gas rather than air was used for catalyst lifting in the Houdry design. This allowed for a higher circulation rate.
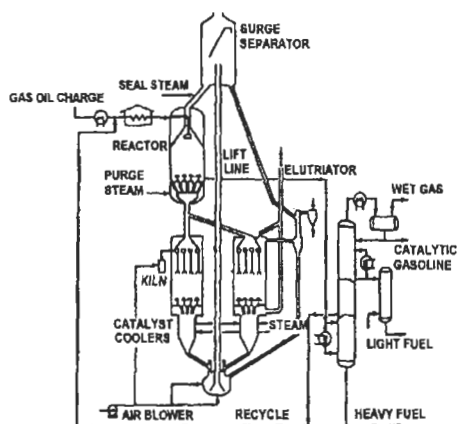
(A)
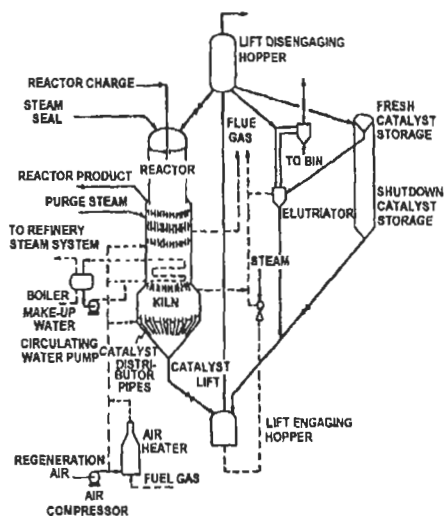
(B)

Figure 19. *Moving bed catalytic crackers: (A) Thermoform moving bed process; (B) Houdry catalytic cracking process.*

The Socony Vacuum design consisted of separate vessels for reaction and regeneration. Units constructed in the late 1940s employed a pneumatic lift design which allowed for high catalyst circulation rates. A typical design is shown in Figure 20, which allowed for a primary air stream to convey the catalyst. A

secondary air stream is injected through an annulus into which the catalyst can flow. By varying the secondary gas flow, the catalyst circulation rate can be varied. This design permitted the injection of all liquid feeds and feeds of higher boiling ranges.

The lift pipe design was tapered to a larger diameter at the top. This minimized the effects of erosion and catalyst attrition, and also prevented the instantaneous total collapse of circulations when the saltation concentration, or velocity, of solids is experienced (i.e. the slump velocity-that velocity below which particles drop out of the flowing gas stream). In a typical operation, 2 % to 4 % coke can be deposited on the catalyst in the reactor and burned in the regenerator. Catalyst circulation is generally not sufficient to remove all the heat of combustion. This facilitated the need for steam or pressurized water coils to be located in the regeneration zone to remove excess heat.

The application of fluidized solids techniques to catalytic cracking resulted in a major technological and process advancement in the petroleum industry. This technology made it possible to transfer all the regeneration heat to the reaction zone. In addition, much larger units could be constructed and heavier liquid feeds could be processed. The development of the fluid solid catalytic cracking process was accelerated by the need for aviation fuel by the Allied nations during World War II. The growth of the fluid-bed process for gasoline and petroleum products manufacture has grown steadily over the years.
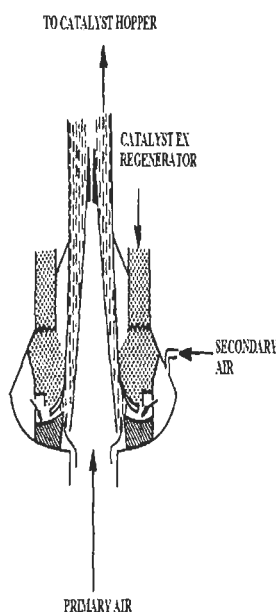
Figure 20. *Catalyst pick-up system for a moving bed catalytic cracker.*

Refinery Applications, Unit Processes and Equipment

Refineries are designed to manufacture products as economically as possible based on the best knowledge available with regard to end product needs, future expansion plans, crude availability and other pertinent factors. A basic modern refinery which does not produce lubricating oils or chemicals is commonly referred to as a fuel products refinery. It is designed to produce primarily motor gasoline, distillate fuels (diesel oil, jet fuel, and heating oil), and bunker (residual) fuel oil. The fuel products refineries can be considered basic and minimum as regards refinery product and processing requirements. Hydroskimming and conversion are the two major variations of this type refinery.

There is a wide range of conversion levels. The term maximum conversion type has no precise definition but is often used to describe a level of conversion, where there is no net fuel oil manufactured. A fuel products refinery with specialities may manufacture lubricating oils, asphalts, greases, solvents, waxes and chemical feed stocks in addition to the primary fuel products. The number and diversity of products will naturally vary from one refinery to another. Refineries produce chemical feed stocks for sale to the chemical affiliates and do not have responsibility for the manufacture of chemical products directly. Both operations may be carried out at the same physical location but the corporate product responsibilities are usually separate.

In addition to the distillation of crude oil coming into the refinery, stills of various designs are used in other types of service throughout the refinery. Cracked products are separated in distillation equipment which is very similar to an atmospheric crude pipe still. The principal difference is that these products are hot from the cracking operation, so that a fired heater is not required.

Catalytic cracking units operate at low pressure, and the primary fractionator operates at the same pressure level as an atmospheric pipe still. Two features of the product vapors affect the design of the lower section of the catalytic fractionator: (1) the vapors are superheated and must be cooled about 200 °F before an appreciable amount of liquid can be condensed, and (2) the vapors from a fluid catalyst unit carry a small amount of fine catalyst particles which might clog the narrow clearances of a conventional bubble cap plate. For these reasons, the bottom section of a catalytic fractionator contains a number of rows of simple baffles. Cool liquid splashes down over the baffles, cooling the vapors, condensing the small amount of bottoms product, and washing the catalyst dust out of the vapor stream. The major part of the liquid leaving the tower bottom is pumped through heat exchangers (which may be steam boilers) and coolers, then returned to the top of the baffled desuperheating section. The flow rate of this circulating reflux stream is often twice as great as the production rate of all the product streams leaving the unit. Normally, all of the heat is removed from the fractionator by several

circulating reflux streams. The proportion of gas and naphtha in the cracked products is much higher than in crude oil, so it is seldom possible to reduce the diameter of the tower top as in atmospheric pipe still design. Due to the low operating pressure, it is necessary to provide expensive compression capacity to permit recovery of these light hydrocarbons in subsequent equipment.

In some situations it is economical to use a single distillation tower to carry out the functions of the cat cracker main fractionator and the fuel vacuum pipe still. This type of tower is called a combination unit. A fuel vacuum pipe still needs large quantities of heat to distill heavy gas oil for cat cracking feed stock. On the other hand, the main fractionator of a catalytic cracking unit has a large desuperheating section for removing excess heat from the cracked products. The combination unit combines two functions in a single tower and uses atmospheric pipe still residuum to desuperheat the cracked products and distill out the valuable gas oil from it. Some operating flexibility is sacrificed and the amount of gas oil distilled is not as great as could be obtained in a vacuum pipe still. This must be balanced against the substantial cost savings involved.

In crude distillation, there are thousands of different compounds present having a virtually continuous spectrum of boiling points. It would be impractical to consider each of these compounds in describing the crude or designing the equipment to process it. Instead the crude is treated as if it were composed of a manageable number (≤ 50) of pseudo components. These are defined by dividing the crude distillation curve into a series of adjacent boiling cuts.

In light ends distillation, by contrast, the number of compounds present is usually less than 30 and may be as low as 3 or 4. Thus, the feed to a light ends distillation tower can be described in terms of the percentages of the various individual compounds present. Contiguous cuts from an atmospheric pipe still have boiling ranges which overlap and there are a large number of compounds which are split between two fractions. Usually the value of the residuum produced is not affected by the quantity of lower-boiling materials it contains, as long as gases and other very light materials have been stripped out.

In light ends fractionation it is usually just as important to remove light material from the heavier cut as it is to keep heavy material out of the lighter cut; sidestreams are seldom withdrawn. The desired purity (expressed as per cent of impurity) of the overhead and bottoms is determined by product specifications or by the requirements of subsequent processing units. To meet these purity requirements, higher reflux ratios and greater numbers of plates between cuts are required than in crude distillation units.

Steam stripping is not adequate for the bottoms purity required. More positive stripping is obtained by charging the tower bottom liquid to the reboiler. In a typical reboiler, 50% of the feed is vaporized and returned to the tower below the bottom plate. A fractionating tower equipped with a steam heated reboiler is shown

in Figure 21. The reboiler may also be heated by a hot oil stream, such as a pumparound reflux stream from the primary fractionator of a cracking unit, or by a fired furnace. Feed to the unit may be all liquid, all vapor, or a mixture of liquid and vapor. Use of a reboiler causes the heavier liquid product to leave the system at a higher temperature than if steam stripping alone were used. For this reason, the use of reboilers is restricted to those systems in which the temperature is well below the level at which cracking takes place. In practice, materials boiling above about 400 °F are seldom distilled in a reboiler type unit.
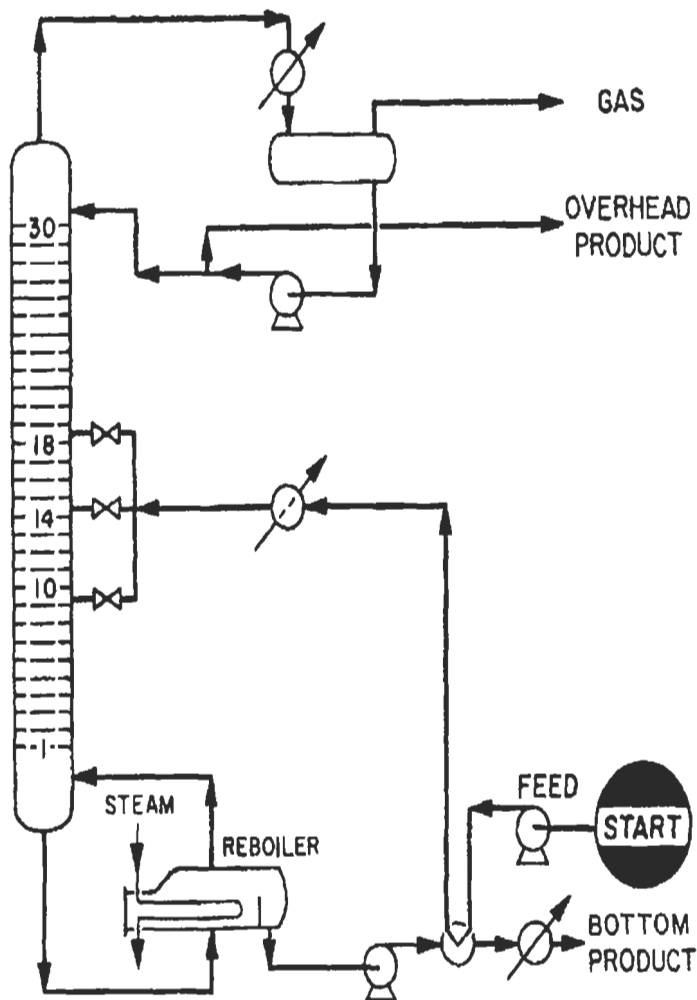
Figure 21. *Typical reboiler operation.*

The overhead products from light ends fractionators are frequently gases, or a mixture of gas and liquid, at normal atmospheric pressure and 100 °F (an average temperature for products condensed by cooling water). To avoid the expense of providing refrigeration to condense product and reflux, operating pressure is maintained at a level that permits the overhead product to be condensed with cooling water. Pressures of 100 to 250 psig are common in refineries; pressures of several hundred pounds are used in oil field plants which recover light naphtha from natural gas. A principal disadvantage of high pressure operation is that the difficulty of separating the products increases with pressure. As mentioned earlier the ease or difficulty of separating two products depends on the difference in their vapor pressures or volatilities. There are situations in the refining industry in which it is desirable to recover a single valuable compound in high purity from a mixture with other hydrocarbons which have boiling points so close to the more valuable product that separation by conventional distillation is a practical impossibility. Two techniques already described which may be applied to these situations are *azeotropic* distillation and *extractive* distillation. Both methods depend upon the addition to the system of a third component which increases the relative volatility of the constituents to be separated.

In azeotropic distillation, the added component is a chemical that forms an azeotrope (constant-boiling mixture) which boils at a lower temperature than any of the constituents of the feed. For example, addition of certain ketones to narrow cut mixtures of paraffins, naphthenes, and an aromatic is known to form azeotropes with the paraffins and naphthenes which boil at temperatures low enough to permit relatively easy separation of them from the aromatic.

In extractive distillation, the compounds which it is desired to separate are distilled in the presence of a solvent. This solvent, by reason of its greater affinity for one of the compounds, causes the other material present to exhibit an abnormally high vapor pressure which permits its removal from the system. For example, adding phenol to a mixture of toluene and close-boiling paraffins and naphthenes causes the toluene to behave as if it boiled as much as 40 °F above its normal boiling point.

Petroleum products may be treated with various solvents for the removal by selective solubility of undesirable constituents or for the recovery of by-products. The solvent and solute must be separated to yield the desired product and to recover the solvent for reuse. The solvents normally boil at a lower temperature than the products from which they are to be removed and so are generally distilled off as overhead products. The pipe stills used for this service may be single-stage or multi-stage units, depending on the service involved. Some solvents can be removed by the use of steam heated stills. In other cases, the high temperature required necessitates the use of fired heaters and vacuum towers.

Rerunning is a second distillation step applied to distillate stocks in order to remove undesirable higher boiling materials from the product. These materials may be

present because of poor fractionation in the primary distillation step; more frequently they are heavy polymers formed in treating operations. Rerunning may be combined with solvent removal, as in the case of heavy lube distillates which are diluted with naphtha to lower their viscosity before being chemically treated. Rerunning operations are characterized by large volumes of distillate products and relatively small residue yields. Frequently, the product is withdrawn as a sidestream with undesirable light fractions passing overhead and polymers being withdrawn from the bottom of the tower. Lube rerun stills usually have several sidestreams which permit close control of flash point and viscosity while producing a wide range of stocks.

Distillation has become an indispensable part of a wide variety of refining processes other than crude distillation. Although the equipment used in these various services differs widely in appearance and complexity, it should be remembered that all distillation operations are fundamentally the same. They all depend on the fact that the liquids to be separated have different boiling points. Whenever such a mixture is heated enough to vaporize part of it, the vapor will have a higher concentration of low boiling point materials than the liquid, while the liquid will be richer in high boiling point materials than the vapor. Another way of stating this is to say that the lower boiling material is more volatile than the higher boiling material. When there is only a small difference in the boiling points of two hydrocarbons (or hydrocarbon mixtures), we say that their relative volatility is low. When their boiling points are widely different, they have a high relative volatility. The higher the relative volatility, the easier it is to separate the components by means of distillation. Thus, the relative volatility of the desired products is an important factor in determining the complexity of the equipment required.

Another major factor affecting the choice of equipment is the boiling range of the feed stock. Crude oil is made up of hydrocarbons varying in boiling point from methane to asphalt, a range of about 1500 °F. The hydrocarbons referred to as light ends (methane through butane) are so low boiling that they are gases at normal atmospheric temperature and pressure. When it is necessary to condense these materials, their boiling point is usually raised by operating the distillation unit under pressure so that available cooling water may be used. In most cases, it would be far more costly to run at lower pressures and condense the light product by means of refrigeration. At the high end of the temperature scale, there are desirable distillate fractions whose atmospheric boiling points are well above the temperature at which cracking starts (roughly 700 °F). To distill these materials without cracking, their boiling points must be lowered by reducing the oil partial pressure. This is accomplished by inducing a vacuum on the system, by diluting the oil vapors with inert gas (usually steam), or by using both methods simultaneously. The stability of the stock with respect to heat determines the maximum temperature which can be employed in the distillation process. The complexity of the process is also affected by the product purity demanded by product specifications or by the

limitations of subsequent processing equipment. The relative values of the several products determine how much money can be spent in designing the unit for increased yields of the more valuable products.

Atmospheric Pipe Stills: A single-stage pipe still used for distilling whole crude is shown in Figure 22. The unit shown is designed to separate six products: gas, overhead distillate, three sidestream distillates, and un-distilled residue, or bottoms. Modern pipe stills usually have several sidestreams. Generally, an atmospheric pipe still is operated at slightly above atmospheric pressure, although pressures as high as 30 psig have been used. The distillate drum temperature is determined by the temperature of the cooling water used in the overhead condenser; normally the drum runs at about 100 °F. The volume of gas produced varies with the operating pressure, the character of the crude, and the boiling range of the overhead distillate. In some cases, all of the vapor leaving the tower is condensed and no gas is produced. The overhead distillate is most frequently a full boiling range naphtha (up to about 400 °F final boiling point) with the first sidestream product being a kerosene or jet engine fuel cut. The second sidestream on the pipe still shown in Figure 22 might be used for diesel fuel or heating oil. The third sidestream might be a heavier diesel fuel or gas oil for feed to a catalytic cracking unit. An atmospheric pipe still is normally designed with enough flexibility to process more than one crude or for more than one distribution of products from a single crude. Additional sidestreams contribute to this flexibility and facilitate the production of narrow boiling range stocks for special uses. The bottoms product in the pipe still shown would probably be blended into heavy fuel oil.
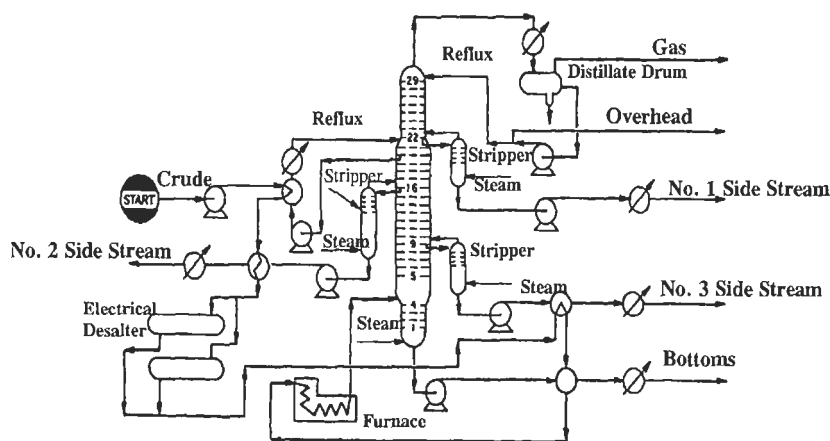
Figure 22. *Single stage atmospheric pipestill.*

The temperature at which the crude enters the tower is usually determined by one of the following considerations:

- When kerosene is one of the distillate products, the color stability of this stock has limited the flash zone temperature to as low as 650 °F in older designs to avoid incipient cracking. Cracking is a function of both time and temperature, and in new designs this limit has been increased by carefully designing the unit to minimize liquid residence time at the high temperature points of the system. If the kerosene product is upgraded in quality by subsequent hydrotreating, the flash zone temperature can be as high as 725 °F or more.

- In single-stage units which do not produce kerosene or other critical stocks, flash zone temperatures may be as high as 750° - 775 °F. The principal limitation is the point at which cracking of distillates to less valuable gas or the rate of coke formation in the furnace tubes becomes excessive.

In two stage units, it is often economical to distill more gas oil in the vacuum stage and less in the atmospheric stage than the maximum attainable. Gas formed in the atmospheric tower bottoms piping at high temperatures tends to overload the vacuum system and thereby to reduce the capacity of the vacuum tower. The volume of crude vaporized at the flash zone is approximately equal to the total volume of distillate products. Of course, the vapor at this point contains some undesirable heavy material and the liquid still contains some valuable distillate products. The concentration of heavy ends in the vapor is reduced by contact with liquid on the trays as the vapor passes up the tower. This liquid reflux is induced by removing heat farther up in the tower.

Reflux from the first tray above the flash zone, along with the flashed crude, enters a smaller diameter stripping section at the bottom of the tower. Here, open steam injected below the bottom plate rises through the descending liquid, stripping out the lower boiling hydrocarbons. These vapors pass up through the flash zone to the fractionating section of the column. Liquid products are withdrawn from plates 9, 16, and 22 as sidestreams. These are contacted with steam in external stripping columns having 4 to 6 plates in order to remove light dissolved hydrocarbons. Since the liquid withdrawn from any plate in a pipe still is in equilibrium with all the lighter vapor passing that plate, it always will contain material which is lower in boiling point than the desired product.

In the case discussed above, part of the heat is removed by returning cold overhead distillate to the top of the tower where it is revaporized by the heat supplied by condensation of part of the rising vapors. The remaining heat is removed by circulating a liquid pump-around stream over plates 20 and 21. This stream is heated by condensation and cooling of the rising vapors, leaves the tower to give up its heat to the feed in an external heat exchanger, then returns to the tower at a higher point. It would be possible to remove all of the heat by pumping cold reflux

from the distillate drum to the top of the tower and thus eliminate the cost of the pumparound circuit. Where more than one sidestream is withdrawn, however, it is usually economical to withdraw part of the heat in a pump-around reflux system farther down the tower. The following economic factors affect the choice:

● Reduction in top reflux permits a reduction in the tower diameter in the section above the pump-around, thereby reducing investment. However, the reduction in reflux requires the addition of more fractionating plates to maintain the same separation efficiency.

● Since temperatures farther down the tower are higher than at the top, heat is available at a higher level. This permits transfer of heat to the incoming feed, thus reducing fuel requirements and furnace investment.

● Cooling water cost is reduced and total heat exchanger plus condenser surface may be reduced.

Steam used in the sidestream strippers and in the stripping section of the main column is condensed in the overhead condenser. This water settles to the bottom of the distillate drum and is drawn off through a small water pot in the bottom. In most installations, ammonia gas is injected into the overhead line to raise the pH of this water and reduce corrosion.

Critical temperatures throughout the tower are controlled by automatic instruments and products are withdrawn under various combinations of flow and level control. A pipe still is capable of running for days on end with only minor adjustment by the operators except, of course, when a change in crude type or in product distribution is required.

Removing Salt from Crude Oil: Crude oil usually contains water which carries inorganic salts in solution. As the crude is heated, the water evaporates and part of the salt is deposited on heat exchanger surfaces, thus impairing their efficiency. To prevent this type of heat exchanger fouling, an electrical *desalting unit* is usually installed as an integral part of the atmospheric pipe still when the crude to be run contains more than 20 to 40 pounds of salt per thousand barrels of oil, or about 0.01 weight per cent. In the desalter, fresh water is mixed with the crude and passed to a coalescing zone. Here a high voltage is applied which causes most of the small water droplets to combine into larger ones. These are separated from the crude in a subsequent settling zone. In some cases, chemicals are added to prevent the formation of emulsions.

Before desalters came into common use, crude pipe stills were frequently equipped with flash drums to minimize salt deposition on hot surfaces. In the flash drum system, the crude is heated to about 300'F. under enough pressure to suppress vaporization. The pressure is released as the crude enters the flash drum and all of the water (along with a small amount of crude) is flashed off, leaving the salt as a

suspension in the oil. The flashed vapor is recombined with the crude near the furnace outlet or in the flash zone of the fractionating tower. While a desalter costs more to install than the flash drum system, it has the advantage of removing up to 95 % of the salt from the oil permanently. Because less salt reaches the fractionating tower in a unit equipped with a desalter, a smaller quantity of corrosion products is formed due to high temperature breakdown and hydrolysis, and the salt content of the residual fuel oil is much lower.

Vacuum Pipe Still: Two rather distinct types of vacuum distillation equipment have evolved in the oil industry. The differences lie in their functions and in details of construction, not in the principles of operation. For convenience, these two types are commonly referred to as lube vacuum pipe stills and fuel vacuum pipe stills. The lube vacuum pipe still was developed in the period between the two World Wars. As the name implies, this type of unit was designed to meet the growing demand for heavy lubricating oil distillates. With this unit, it is possible to produce distillates which boil as high as 1050°-1100 °F at atmospheric pressure. The lube vacuum pipe still is similar in many ways to the atmospheric pipe still previously described. The reduced crude feed (or whole crude, in an integrated two-stage pipe still) is preheated by indirect exchange with the hot product streams, then flows through the furnace and transfer line to the flash zone. The flash zone operates at an absolute pressure of 125-150 millimeters (mm) of mercury. Liquid from the flash. zone is stripped with steam to increase distillate yield and steam is sometimes injected into the heating coil also. By lowering the boiling point of the feed, coil steam may reduce the peak coil temperature required for a given yield, thus minimizing gas formation and product degradation.

Cracking imposes an additional penalty in a vacuum unit in that it forms gas which cannot be condensed at the low pressures employed. This gas must be vented by compressing it to atmospheric pressure. This is accomplished by means of steam jet ejectors. Ideally, it would be possible to operate a vacuum pipe still without ejectors, with the overhead vapors composed only of steam. In practice, however, leakage of air into the system and the minor cracking which occurs make it necessary to provide a means of removing non-condensibles from the system. In addition to the distillation of atmospheric residuum, the lube vacuum pipe still is also used for rerunning of off specification lube distillates.

The fuel vacuum pipe still is actually a simplified model of the lube unit, rather than a distinct breed of its own. This type of still did not come into general use until shortly after World War II. As demand for high quality motor gasoline soared in the immediate postwar period, refineries began digging deeper into the crude to augment the supply of cat cracking feed stocks. The lube vacuum pipe still was not ideally suited to this goal because it was designed to prepare several well fractionated lube distillates. The large number of plates needed for fractionation imposes a relatively large pressure drop between the condenser and the flash zone; the resulting high flash zone pressure reduces the yield of heavy gas oil which can be obtained at a given temperature. To obtain a low flash zone pressure, the number of plates in the upper section of the vacuum pipe still is reduced to the minimum

necessary to provide adequate heat transfer for condensing the distillate with the pumparound streams. A section of plates is included just above the flash zone. Here the vapors rising from the flash zone are contacted with reflux from the product drawoff plate. This part of the tower, called the wash section, serves to remove droplets of pitch entrained in the flash zone and also provides a moderate amount of fractionation. The flash zone operates at an absolute pressure of 60-90 mm Hg. Another variation in fuel vacuum pipe still designs is to use contacting devices that give lower pressure drop than distillation trays. These permit a lower flash zone operating pressure and thereby allow higher yields of heavy gas oil. The plates in the wash section are replaced with Glitsch grids. These grids are highly efficient de-entrainment devices and give a reasonable fractionation contacting efficiency. The plates in the pumparound sections are replaced with Pall ring packing. This packing competes well with plates in providing the required heat transfer area in the minimum practical tower height.

Since cracking stocks generally do not have to meet the color specifications that lube distillates do, higher flash zone temperatures (up to 800 °F) can be tolerated. Fuel units are normally designed to distill material boiling up to 1,100 °F (at atmospheric pressure) from the feed, and some units have distilled beyond 1,200 °F at low feed rates. The fuel vacuum pipe still is also used to recover cracked gas oil from the tar formed in residuum cracking (visbreaking) processes. In this service, it is frequently referred to as a *vacuum flash unit*. Pipe stills designed for the production of asphalt are usually the fuel type of unit.

Hydroskimmer: A hydroskimming refinery lends itself to locations where the market demands for the major fuel products (gasoline, gas oil, and residual fuel oil) approximate the quantities of these products obtainable by distillation from the available crudes. A typical hydroskimming refinery would include an atmospheric pipestill, powerforming (catalytic naphtha reforming), light-ends recovery and fractionation, and treating and blending. The atmospheric pipestill performs the initial distillation of crude oil into gas, naphtha, distillates, and residuum. The naphtha may be separated into gasoline blending stock, solvents, and powerformer feed. The distillates include kerosene, jet fuel, heating oil and diesel oil. The residuum is blended for use as bunker fuel oil. The powerforming unit is required to upgrade virgin naphtha to produce high octane gasoline. Powerforming is a fixed bed catalytic reforming process employing a regenerable platinum catalyst. In the process, a series of reactions takes place. The most important of these is aromatization; other reactions include isomerization, cracking, hydrogenation, and polymerization. The desired product is of approximately the same boiling range as the feed, but the molecules have been rearranged or reformed into higher octane compounds. Light ends recovery and fractionating equipment is necessary after the Powerformer and on the pipestill overhead stream to separate the effluent mixtures into the desired boiling range cuts. Hydrofining is used to reduce sulfur and/or other impurities and to improve odor, color, and stability of the pipestill fractions. Hydrofining is a fixed-bed catalytic process using a regenerable cobalt molybdate

catalyst in a hydrogen atmosphere. The hydrogen is produced by the Powerformer with supplemental hydrogen manufactured if necessary. The difficulty of hydrofining (desulfurization) increases with increase in the hydrocarbon boiling point. Naphthas are generally desulfurized up to 99 + % by hydrofining while the maximum desulfurization of distillates is usually 90 %. The components produced by the process sequence outlined above are blended as required to meet final product rates and qualities. The hydroskimming type refinery is used where the gasoline demand is substantially lower and hence the final product demand is close to that yielded by single stage distillation operations. In those areas where the demand for gasoline is relatively high, conversion processing is required.

In a typical conversion type refinery the atmospheric P/S residuum can be fed to a vacuum pipestill. The vacuum tower enables the refiner to cut deeper into the crude, at the same time avoiding high temperatures (above about 750 °F) which cause thermal cracking with resultant deposition of coke and tarry residues in the equipment. The vacuum gas oil produced by vacuum distillation is fed to a catalytic cracking unit for conversion into high octane gasoline blending stock. Byproducts are gas, distillate, cycle gas oil, and fractionator bottoms. The process uses a fluidized catalyst system. The catalyst is circulated continuously between the reactor where cracking takes place and the regenerator where the coke deposited on the catalyst is burned off. The major competing process is hydrocracking which offers greater conversion and flexibility but usually requires a higher investment.

Hydrocracking is a fixed bed catalytic process which cracks and hydrogenates hydrocarbon feeds. The process consumes large quantities of hydrogen and a hydrogen plant is usually necessary to support the operation. Practically any stock can be hydrocracked, including refractory feeds which resist conversion by other processes. In general, the very heavy residuum from the vacuum pipestill does not make good quality feed for catalytic cracking. In the refinery shown it is blended into residual fuel oil. Many times, however, the market for large volumes of residual fuel oil does not exist. When this is the case, additional conversion units are added to further process the vacuum pipestill bottoms. In other words, the higher the conversion of the refinery the more lighter fractions are produced. The relative levels of conversion vary from refinery to refinery.

In a typical maximum conversion type refinery, the higher conversion levels are obtained by additional processing of the bottoms and/or light ends. To increase conversion of the bottoms the amount and/or severity of processing is increased. The resulting fuel oil levels may decrease to zero. Included here in addition to the basic components of a conversion refinery may be fluid coking, delayed coking, and/or visbreaking. These processes are basically thermal cracking processes for reducing the volume and viscosity of the vacuum residuum while producing appreciable quantities of lighter products. Each of the three processes is commercially used with selection based on particular needs at a given refinery. Some of the various characteristics include:

- Coking-Delayed Coking and Fluid Coking are the two major variations of this process. Fluid coking produces less coke as compared with delayed coking and hence yields a better product distribution. That is, for a given product slate less crude is converted into coke. The coke produced by fluid coking, however, is of little value as it consists of fine hard particles in contrast to large pieces for delayed coke. This difference in size and texture is important to electrode manufacturers who historically have used delayed coke.

- Visbreaking is the least expensive of the cracking processes but is limited to the lowest conversion of perhaps 20 to 25 % of the feed to 680 °F material. To obtain light ends conversion, alkylation and polymerization are used to increase the relative amounts of liquid fuel products manufactured. The process of alkylation is used to convert olefins, (propylene, butylenes, amylenes, etc.), into high octane gasoline by reacting them with isobutane. Polymerization involves reaction of propylene and/or butylenes to produce an unsaturated hydrocarbon mixture in the motor gasoline boiling range.

An old variation of the conversion type is a catalytic combination unit. Development of this scheme was necessitated by the rising cost of refinery construction after World War II and by the great demand for capital for postwar expansion. The scheme reduced the investment and operating costs for refining equipment. The basic feature of the combination unit lies in the integration of the fractionation facilities of the reduced crude distillation and catalytic cracking sections.

Fuel Products with Specialties

A fuel products refinery with specialties may manufacture products such as lubricating oils, asphalts, greases, solvents, waxes and chemical feed stocks in addition to the primary fuel products. The number and diversity of products will naturally vary from one refinery to another, but for purposes of discussion a fuel products refinery with specialties may include many of the following processes.

Two-Stage Crude Distillation (Atmospheric and Vacuum): The vacuum stage can be used alternately to produce heavy gas oil for catalytic cracking feed or raw lube distillate cuts for lubricating oil manufacture.

Virgin Naphtha Catalytic Reforming (Powerforming): This technique is used for the production of high octane motor gasoline, or as a source of aromatic compounds.

Light Ends Recovery, Fractionation, and Conversion: Propylenes and butylenes may be recovered for feed to a polymerization plant for production of high octane

gasoline, or chemicals. Butylenes and isobutane may be desired for use in an alkylation plant where they are combined to make aviation gasoline and motor gasoline blend stocks. Propanes and butanes may be recovered in essentially pure form for sale as liquefied petroleum gases. It may be profitable to recover ethylene for chemical production. Certain of the light ends components, particularly ethylene, propylene, and butadiene are so in demand that processes such as steam cracking are employed specifically for their production.

Fuel Products Treating: (a) Sweetening: This is a process for improving odor of gasolines, kerosene, and heating oils. The foul smelling mercaptans are converted into bisulfides whose odor is much less objectionable. Among the types in use are copper chloride, hypochlorite, Merox, and air inhibitor sweetening. (b) Hydroprocessing - The nomenclature system with regard to hydrogen processing is quite confusing with an array of labels involving trade names, terms such as mild, medium, and severe, high and low pressure. Choice of terminology varies widely from company to company. A wide variety of petroleum fractions may be treated at elevated temperature and pressure with hydrogen in the presence of a catalyst to reduce sulfur, improve stability, odor, combustion characteristics, appearance, and to convert heavy fractions to lighter more valuable products. The most severe form of hydroprocessing as discussed previously is hydrocracking. For fuel products treating, however, two less severe hydroprocessing operations are used, hydrofining and hydrotreating. Hydrofining usually involves only minor molecular changes of the feed with hydrogen consumption in the range of about 100 to 1,000 cu.ft./bbl. Typical applications include desulfurization of a wide range of feeds (naphtha, light and heavy distillates, and certain residua) and occasional pretreatment of cat cracker feeds. Hydrotreating essentially involves no reduction in molecular size with hydrogen consumption less than about 100 cu. ft./bbl. Primary application is to remove small amounts of impurities with typical uses including naphtha and kerosene hydrosweetening. (c) SO_2 Extraction - This is a method of solvent extraction with liquid SO_2 to remove aromatic hydrocarbons and cyclic sulfur compounds. It is used to improve the burning qualities of kerosene and diesel fuels, and to reduce sulfur. This process has practically been supplanted by other solvent extraction or by hydrotreating.

Residuum Conversion: This includes fluid coking, delayed coking, visbreaking, and residuum hydroprocessing.

Solvent Deasphalting: This is the solvent extraction of virgin residuum to remove asphaltenes or other tarry constituents. The deasphalted oil may be further processed into lubricating oils and greases, or used as cat cracking feed.

Lubricating Oil Manufacture: This consists of solvent deasphalting, phenol treating, and solvent dewaxing. In dewaxing, waxy lube is diluted with a solvent such as propane or methyl ethyl ketone (MEK), and cooled to crystallize the wax which is then removed by filtration.

Grease Manufacture: Selected lube oil fractions are blended with various metallic soaps to produce high viscosity lubricating greases.

Wax Manufacture: A waxy distillate cut from crude or the wax byproduct from lube oil dewaxing is first deoiled. Resulting low oil content wax is hydrofined for color improvement and fractionated into appropriate melting point grades.

Asphalt Manufacture: Saleable asphalts are produced from the residua of selected crudes. The residuum itself may be sold as straight reduced cuts to make it easier to handle, producing the so called cut-back asphalts. Another variation is air blown or oxidized asphalts for improved tenacity, greater resistance to weathering, and decreased brittleness. Emulsified asphalts are made for application at relatively low temperatures.

Chemical and Other Specialty Manufacture: A wide variety of products may be derived from petroleum feed stocks, including such diverse materials as alcohols, butyl rubber, sulfur, additives, and resins. Other specialties such as solvent naphthas, white oils, Isopars, Varsol, may also be produced. As indicated previously the respective chemical affiliate usually has responsibility for products broadly classified as petrochemicals.

Fluid catalytic cracking and hydrocracking are two additional processes that are often encountered. There are many other processes used in refineries not mentioned here. The list above is intended only to emphasize the wide diversity of processing which is common to petroleum refining and to introduce in a very general way some of the more important of these processes. Also it must be emphasized that only fundamental principles of refinery operations have been discussed and modern manufacturing techniques vary widely from company to company.

PRODUCTS FROM PETROLEUM

The following section provides general information on the major products and processes used to manufacture them from crude oil. The basic refinery operations have already been described. Emphasis is now given to the downstream processes which are used in transforming distillates into a multitude of consumer products. Not all products and processes are discussed, and indeed, only highlights are provided on those discussed.

Light Ends Conversion Processes and Products

In this subsection we will concern ourselves with those light ends processes which effect a chemical change in the hydrocarbon mixture: alkylation, polymerization,

and isomerization. Alkylation is a catalytic reaction involving the addition of a molecule of isobutane to an olefin. In the conventional alkylation process, isobutane is added to propylene, butenes, or amylenes to produce high octane aviation gasoline components. A typical reaction which occurs in alkylation is illustrated in Figure 23.

Polymerization is a reaction in which several molecules of the same or similar material combine to form a larger molecule. We will only discuss the polymerization of C3, C4, and C5 olefins to products in the gasoline boiling range (C5 - C12). A typical reaction which occurs in polymerization is illustrated in Figure 24.

The principal use of the alkylation process is the production of high octane aviation and motor gasoline blending stocks by the chemical addition of C2, C3, C4, or C5 olefins or mixtures of these olefins to an iso-paraffin, usually isobutane. Alkylation of benzene with olefins to produce styrene, cumene, and detergent alkylate are petrochemical processes. The alkylation reaction can be promoted by concentrated sulfuric acid, hydrofluoric acid, aluminum chloride, or boron fluoride at low temperatures. Thermal alkylation is possible at high temperatures and very high pressures.
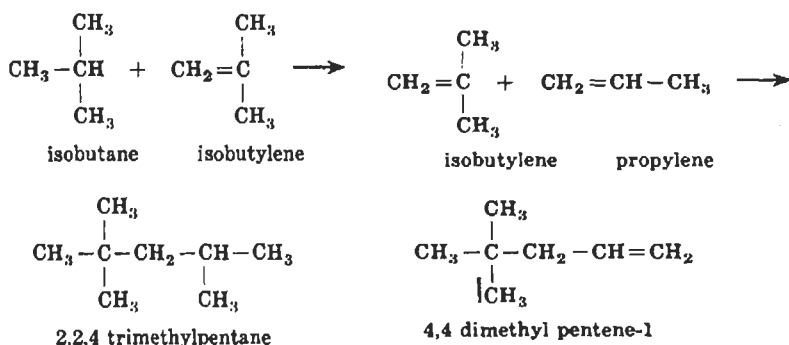
Figure 23. *Alkylation reaction.*

Figure 24. *Polymerization reaction.*
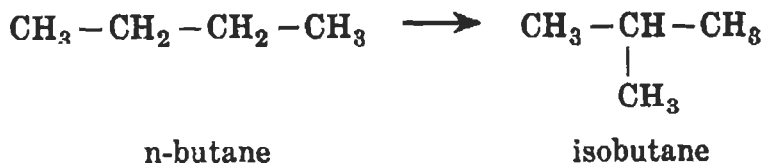
Figure 25. *Isomerization reaction.*

In conventional alkylation operations, 98 wt. %, sulfuric acid is used as the catalyst, although some processes use HF. The spent alkylation acid, withdrawn as 88-92% acid, is not consumed in the chemical sense, but is diluted by carbonaceous material and small amounts of water. Acid reconditioning is usually completed in a separate plant. The range in makeup acid requirement and in octane quality varies with plant design, with type of feedstock, and with alkylate product rate. A wide variety of feedstocks can be processed through alkylation plants, as both low and high boiling olefins can be alkylated.

With sulfuric acid the reaction is usually restricted to propylene, butylenes, or pentylenes as the feed olefin, with C4 = 's the most frequently used due to the high octane number of the resultant alkylate. Isobutane is the preferred isoparaffin in the reaction. It may be obtained from crude oil, catalytic cracking, hydrocracking, Powerforming, or by isomerization of normal butane. The alkylation process is normally operated for the maximum yield of product in the range of C6 to 350 ° FVT. Basically, the alkylation process is operated for the maximum yield of high octane number paraffin isomers such as trimethylpentanes, which have the highest octane rating of all isoparaffinic C8 hydrocarbons. A typical alkylation plant is schematically shown in Figure 26. The alkylation plant proper consists of the feed preparation section, the reaction and refrigeration sections, and the fractionation section. The olefin feed stream is caustic washed to remove sulfur compounds and is chilled. Entrained water is then removed by coalescing and the olefin enters the reactor compartments through a multiplicity of small jets. Since normally the olefin feed does not contain enough isobutane to completely alkylate the olefins present, field butanes provide additional isobutane.
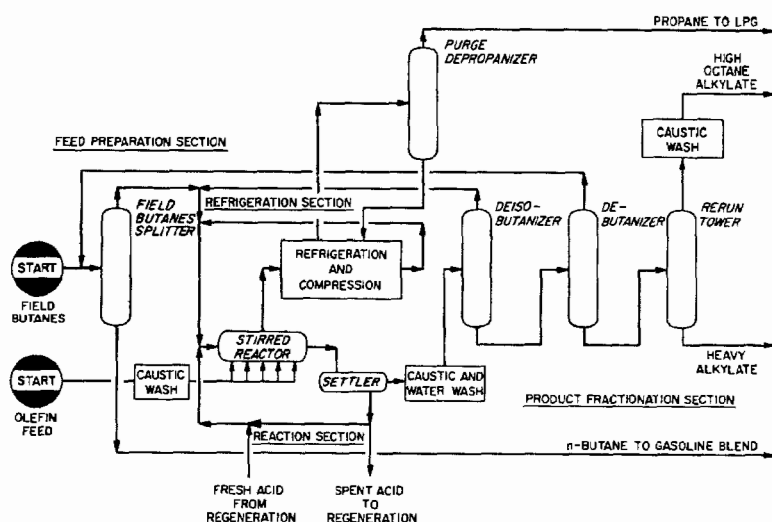
Figure 26. Example of an alkylation plant.

The field butanes splitter serves to concentrate the isobutane in this stream; most of the normal butane is removed in the splitter bottoms for use in gasoline blending. The isobutane rich overhead is joined by a similar stream from the deisobutanizer tower in the Fractionation Section. The combined stream is cooled and dried and then is mixed with H_2SO_4 acid catalyst recycled from the settler before proceeding to the reactor. The reaction occurs at 35-60 °F in a series of vigorously agitated mixing compartments through which the isobutane rich acid emulsion moves in series flow. The exothermic heat of reaction is removed by autorefrigeration resulting from vaporization of a portion of the reaction mixture. The reactor effluent emulsion is broken in the settler, providing a product hydrocarbon stream for fractionation and an acid stream for recycle to the reactor. Compression in the refrigeration sections permits heat removal from the system to be accomplished by cooling water. A portion of the iC4 rich refrigerant is sent to a purge depropanizer to avoid excessive buildup of C3 in the system. The settled reactor hydrocarbon is caustic and water washed before proceeding to the deisobutanizer where iC4 is taken overhead for recycle to the reactors. Additional removal of C4 from alkylate is accomplished in the debutanizer before the alkylate is cut to the desired end point in the rerun tower. The rerun alkylate is usually caustic and water washed before going to blending. Debutanizer overhead can be recycled back to the splitter tower to recover additional isobutane, thus reducing field butane requirements. Light olefins can be polymerized to products in the gasoline boiling range by passage over a phosphoric acid catalyst at 400 to 500 °F. and 500 to 1000 psig. In one type of process, the phosphoric acid is supported on kieselguhr clay pellets. The catalyst becomes carbonized or softens and crumbles as a run progresses and has to be replaced when the pressure drop through a reactor becomes excessive, and/or catalyst activity has been lost. The first type of process is the "selective" operation for the production of aviation gasoline base stock. In this operation, isobutylene alone or a mixture of butylenes is polymerized at about 300 - 350 °F to form some multiple of C4. The selective polymer is then hydrogenated to form a high ASTM octane number branched paraffin (2,2,4-trimethyl pentane; 2,2,3-trimethyl pentane, etc.). The more common type of catalytic polymerization is the "non-selective" operation which is a high conversion process aimed at the production of motor gasoline by polymerizing either propylene, mixed butenes, or both. Since they are practically pure mono-olefins, narrow fractions of the polymer product are often valuable chemical raw materials. Examples are C7 and C9 cuts for Oxo alcohol manufacture, and C12 (propylene tetramer), which is used in the production of detergent alkylate. In a typical non-selective polymerization plant, the feed to the plant is a mixed C3 - C4 stream from a catalytic light ends plant debutanizer. The products are a C3 stream which is fed to the LPG plant; a C 4 stream for blending purposes; the polymer gasoline product; and heavy polymer which goes to refinery slop. The usual poly plant consists of three sections: feed preparation, reaction, and product fractionation. An example of such a plant is shown by the flowsheet in Figure 27.
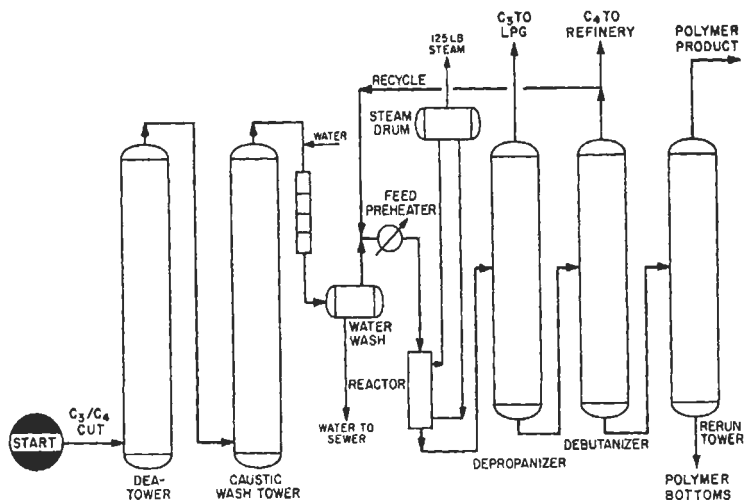
Figure 27. *Example of a ploymerization plant.*

In the feed preparation section, those materials are removed from the reactor feed which would either poison the catalyst or which would give rise to compounds detrimental to product quality. Hydrogen sulfide is removed in the DEA tower, and mercaptans are taken out in the caustic wash. The water wash removes traces of caustic and DEA, both of which are serious catalyst poisons. Also, the water wash is used to control the water content of the reactor feed (which has to be kept at a predetermined level to keep the polymerization catalyst properly hydrated) and remove NH_3, which would poison the catalyst. Diolefins and oxygen should also be kept out of poly feed for good operation.

The reaction section consists of the high pressure reactors filled with catalyst, and means to take away or dissipate the high heat of reaction (300-500 Btu/lb of olefin polymerized). In the tubular reactors, the catalyst is inside a multiplicity of tubes which are cooled by a steam-water condensate jacket. Thus, the heat of reaction is utilized to generate high pressure steam. In the chamber process, the catalyst is held in several beds in a drum-type reactor with feed or recycled product introduced as' a quench between the individual beds.

The fractionation section consists of the towers required to separate the unconverted light ends from the polymer gasoline on the one hand, and the polymer gasoline from the heavy polymer on the other hand. This latter operation, referred to as "rerunning", is occasionally required to meet gasoline product specifications. Usually, poly plants are designed for 90-95% conversion, and an average catalyst life of about 100-150 gals/lb. The reactors are generally operated at 375 to 400 °F, and 500 to 1,000 psig and a space velocity of 0.25 to 0.35 gals. feed/lb of catalyst/hr. The rate of polymerization decreases in the following order:

isobutylene, butene-1, propylene, butene-2. However, the inclusion of butylenes with a propylene stream can increase the propylene conversion. Increases in temperature or feed olefin concentration shorten catalyst life. Recycle is employed where necessary to dilute the olefins in the feed, thereby reducing the temperature rise and lengthening catalyst life.

The isomerization process was developed originally for converting normal butane to isobutane for alkylation. Processes were later developed for increasing the octane of pentane and hexane streams by isomerization. The historical catalyst used is solid aluminum chloride supported on bauxite. Hydrogen chloride gas is used as a promoter to increase reaction speed. When used in conjunction with an alkylation plant, an isomerization plant is fed spent butane from the alkylation fractionation system. This type of feed is suited for processing in an isomerization plant because it is essentially pure normal butane. Naturally-occurring butane can also be used as a feedstock but sulfur and olefin content must be very low. Figure 28 shows the schematic flow through a liquid phase butane isomerization plant. The normal butane feed (olefin and sulfur free) is first dried and preheated to about 200 °F. A fraction of the feed is passed through a catalyst makeup drum where the butane dissolves a small quantity of the catalyst for makeup purposes. The major portion of the hot feed is joined by a recycle HCl stream from the HCl stripper tower. A small quantity of hydrogen is formed in the reaction and this is removed in the vent gas scrubber. The feed (now containing about 5 mol% HCl to promote the reaction) proceeds to the reactors. The reactor vessels contain beds of porocel (low iron content bauxite) which absorbs the $AlCl_3$ on its surface to give an active catalyst of long life. $AlCl_3$ loss from the reactor vessel is negligible.
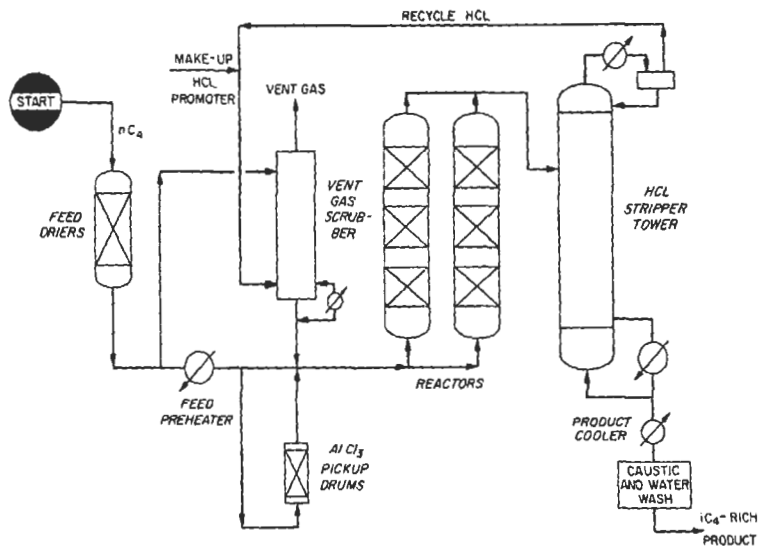
Figure 28. *Example of an isomerization plant.*

After reaction at 200 - 250 °F and 350 psig the reactor effluent is stripped to remove recycle HCl. The stripper bottoms is cooled and caustic washed to remove remaining traces of HCl. The product can then be sent to the alkylation plant for fractionation or a tower provided in the isomerization plant for fractionation of isobutane and recycle of unconverted normal butane back to isomerization.

Lubricating Oils

Lube oil distillates are composed mainly of normal paraffins, isoparaffins, naphthenes and aromatics. Also present in smaller amounts are certain organic compounds containing oxygen, sulfur and nitrogen which are impurities. The predominant molecular structures in lube distillates are naphthene rings or naphthene-aromatic rings arranged in groups of one to as many as six rings. To these ring structures, paraffin chains are attached. The more such chains, the more paraffinic in character will be the oil. Naphthenic oils generally have more naphthene-aromatic rings and fewer paraffin side chains attached to them. Normal paraffins and high molecular weight isoparaffins, which have a high melting point (i.e., over 130 °F) give the unprocessed lube fractions their waxy character and a typical pour point of about 40- 110 °F. These normal paraffin waxes must be removed from the oil before it can be used. Isoparaffins present in the oil are partially removed during the paraffin waxes removal, but those remaining do not affect the pour point of the oil. Naphthenic lube distillates are very different from paraffinic lube distillates both in composition and quality. Crude oil is a complex mixture of hydrocarbons. This mixture includes gases with boiling points well below 0 °F as well as liquids and heavy "pitch" (asphalt) components with boiling points higher than 1,100 °F. The crude oil components are separated into various boiling range fractions or "cuts" by distillation. Distillation of a crude oil is first carried out at atmospheric pressure. The bottoms from the atmospheric distillation tower or "reduced crude" is the fraction from the crude oil containing the lubricating oil fractions together with wax and asphalt. Reduced crude components usually have boiling points higher than 650 to 700 °F. The reduced crude is then distilled under vacuum to obtain three or four narrow boiling lube fractions, an overhead stream or vacuum gas oil and a bottoms stream or vacuum residuum. The vacuum residuum is also used to produce high viscosity lubes. Vacuum distillation is thus the first step in the manufacture of lube oils. Distillation of the reduced crude must be carried out under vacuum to prevent thermal cracking and coking which would occur at the normal boiling point of lube oil fractions. Generally lube distillates are cut to viscosity and boiling range specifications in the vacuum distillation tower, which is called the vacuum pipestill (VPS).

Lube refining consists of several treating steps. Lube oil fractions as obtained from the VPS are seldom suitable as base stocks for blending branded lubricants or

specialty products. Manufacture of lube base stocks from the vacuum distillates is usually accomplished in several consecutive processing operations. The specific lube processing required is a function of the finished base stock specifications and feed stock qualities. Each lube process is aimed at improving one quality of the base stock in order to meet the corresponding finished base stock specification. Along with this main effect, each process has secondary effects on several other base stock qualities. These secondary effects need not necessarily be improvements to the quality of the base stock. Viscosity change is the most important secondary effect for all processes. The net viscosity change across processing and the finished base stock viscosity specification will determine the viscosity of the distillates cut at the VPS.

Solvent extraction is one of the most important steps in the manufacture of lubes. This is a physical separation process that uses a solvent to preferentially dissolve and remove from lube distillates aromatic compounds which cause a large change of viscosity with temperature. Viscosity index (VI) of an oil is an arbitrary relative measure of its change in viscosity with temperature. The smaller the change in viscosity of an oil with a given change in temperature the higher the VI of the oil. A high VI is desirable in high quality motor oils. The amount of material to be extracted depends upon the increase in VI required.

Hydrofining is a catalytic fixed-bed process that utilizes hydrogen as a treating agent to remove sulfur and nitrogen and to partially saturate hydrocarbon molecules, thereby improving color and color stability of the oils. Reactions that occur in hydrofining also reduce the organic acidity (i.e., neutralization number), Conradson carbon and viscosity of the lubes. Solvent dewaxing is a physical separation process in which wax is crystallized at low temperature from a mixture of waxy oil and solvent. The solvent is used to provide favorable conditions for growth of wax crystals and to reduce the viscosity of the resultant slurry so as to permit continuous dewaxed oil/wax separation using a rotary filter. By dewaxing the natural pour point of the oil is lowered, making flow of the oil at lower temperatures possible. Dewaxing is a necessary step in the manufacture of most paraffinic lubes. Solvent deasphalting is a physical separation process used to recover high viscosity lube oil fractions from the vacuum residuum. When mixed with the residuum, the solvent (usually propane) preferentially dissolves the oil and precipitates asphalt and resins. It would not be possible to separate this very high viscosity lube from the asphaltic materials in a vacuum pipe-still due to its high boiling point (i.e., above 1,100 °F). Deasphalting makes the separation possible at low temperatures, thus eliminating the possibility of thermal cracking and coking. The lube hydrocracking process is a hydrotreating process which is much more severe than the hydrofining technique described previously and can be used to replace solvent extraction for improving the viscosity index of raw distillates or deasphalted oil. This process was first employed in the United States in the period of 1932-1936 to make lubes having VI's of 90-100.
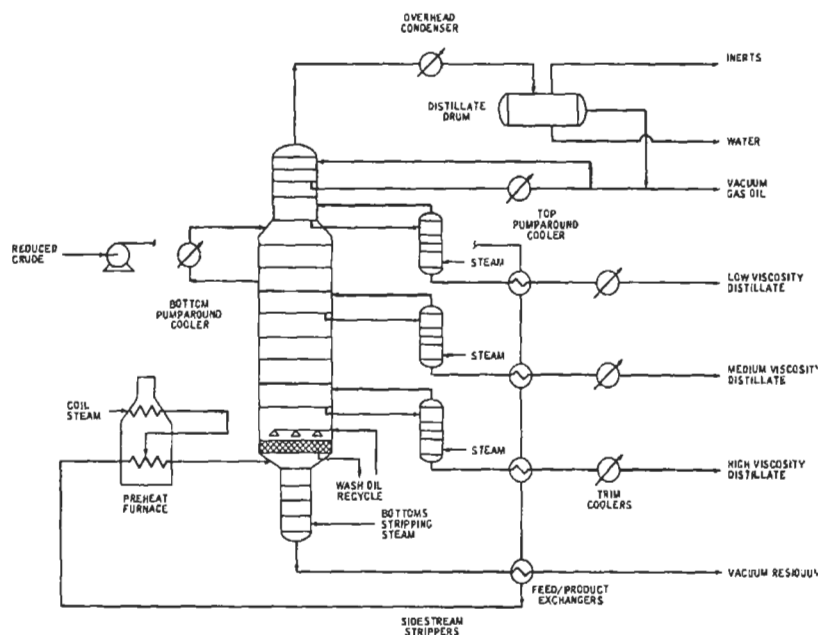
Figure 29. *Example of a lube vacuum pipestill.*

As noted earlier, vacuum distillation is the first step of lube oil manufacture. By distillation the reduced crude is cut into fractions for the production of light, medium and high viscosity lube base stocks. It is important that lube fractions of the correct viscosity and boiling range be cut at the VPS, since subsequent lube processing operations cannot be used to control the values of these two properties in the finished lube products. A simplified process flow diagram for a typical lube VPS is illustrated in Figure 29. In a vacuum distillation process, the reduced crude from tankage is preheated by exchange with the hot sidestreams and vacuum bottoms products. The preheated VPS feed is then partially vaporized in a furnace where its temperature is raised to about 750 °F. Superheated steam (referred to as "coil steam") is injected into the furnace tubes to lower the hydrocarbon partial pressure thus reducing the furnace outlet temperature required for a given degree of vaporization. Coil steam injection also prevents thermal cracking by decreasing residence time in the furnace tubes.

The heated oil is flashed into the VPS flash zone where vapor and liquid separate. Split between distillate and bottoms depends on flash zone temperature and pressure. Separation of vapor and liquid in the flash zone is incomplete, since some lower boiling sidestream components are present in the liquid while bottoms components are entrained in the vapor. The liquid from the flash zone is steam stripped in the bottom section of the VPS to remove the lower boiling components.

The vapors from the flash zone are cleaned from bottoms components in the VPS "wash zone," which is located immediately above the flash zone. The VPS wash zone consists simply of an entrainment removal device called a Glitsch grid which works on the same principle as a crinkled wire mesh screen. This device provides a large amount of surface area on which entrained bottoms components can coalesce. The coalesced liquid is then washed from the grid by clean wash oil distributed uniformly over the grid by means of spray nozzles. Coalesced liquid and wash oil from the wash zone are removed using a total drawoff tray. Vapors passing overhead from the VPS wash zone are fractionated and condensed in the upper section of the VPS. Liquid reflux is necessary in this section of the tower to obtain the desired degree of fractionation.

Internal reflux is induced by means of externally cooled liquid pumparounds. A pumparound simply removes hot liquid from the tower, pumps it through a heat exchanger and then introduces this cooled liquid into the tower a few trays above. Use of pumparounds allows a better distribution of tower loadings than if all the heat were removed from the VPS using an overhead condenser. Four to six trays between sidestreams and two pumparounds are normally specified for a lube VPS. The three liquid sidestream products to be used as lube plant feed stocks are steam stripped to remove lighter boiling components which condense with the sidestreams.

The VPS overhead consists of steam, inerts, condensable and non-condensable hydrocarbons. The condensables result from low boiling material present in the reduced crude feed and from entrainment of liquid from the VPS top tray. The non-condensables result from cracking at the high temperatures employed in the VPS. Inerts result from leakage of air into the evacuated system. Steam and condensable hydrocarbons are condensed using an overhead water-cooled condenser. The distillate drum serves to separate inerts and non-condensables from condensate, as well as liquid hydrocarbons from water. Vacuum is maintained in the VPS using steam jet ejectors.

Lube oil extraction plants often use phenol as solvent. Phenol is used because of its solvent power with a wide range of feed stocks and its ease of recovery. Phenol preferentially dissolves aromatic-type hydrocarbons from the feed stock and improves its oxidation stability and to some extent its color. Phenol extraction can be used over the entire viscosity range of lube distillates and deasphalted oils. The phenol solvent extraction separation is primarily by molecular type or composition. In order to accomplish a separation by solvent extraction, it is necessary that two liquid phases be present. In phenol solvent extraction of lubricating oils these two phases are an oil-rich phase and a phenol-rich phase. The oil-rich phase or raffinate solution consists of the "treated" oil from which undesirable naphthenic and aromatic components have been removed plus some dissolved phenol. The phenol-rich phase or extract solution consists mainly of the bulk of the phenol plus the undesirable components removed from the oil feed. The oil materials remaining

from the raffinate and extract solutions after the phenol solvent has been recovered are called the raffinate and extract oils, respectively. In solvent extraction of lube oils, the solvent is said to be "selective" with respect to the undesirable components, which are more soluble in it. The more selective the solvent, the sharper the separation between desirable and undesirable oil components.

Asphalt

A straight reduced asphalt is the residual product after removing the most volatile or lower boiling components (usually lower than 1,000 °F normal boiling point) from crude oil. Crude oils vary in their content of asphalt residuum, and may contain as little as 1 LV% or as much as over 60 LV%. From a given crude oil the asphalt yield will vary with the consistency (i.e., penetration) of the residuum, which in turn is dependent upon the extent of removal of the lower boiling components. Thus, the lower the penetration of the asphalt residuum, the lower is its yield on crude. There are three general methods by which asphalts of this type can be manufactured. These methods are vacuum distillation, blending and solvent separation. Among these methods vacuum distillation has historically been the most popular method for the manufacture of straight reduced asphalts. After distillation of the crude oil in an atmospheric pipestill, the reduced crude is partially vaporized in a furnace and continuously flashed in a vacuum pipestill (VPS). Straight reduced asphalt is obtained as bottoms from the VPS, whereas a "broad cut" vacuum gas oil (VGO) is obtained as overhead from the VPS. Penetration of the vacuum residuum will be determined primarily by the temperature and pressure at which the reduced crude is flashed in the VPS. Vacuum reduced asphalts typically have initial boiling points of about 1,000 °F (atmospheric equivalent boiling point), although this depends on the inherent composition of the crude oil. The VGO can be used for "fluxing" (i.e., softening) certain straight reduced asphalt grades for specific applications or as cracking feed stock. Straight reduced asphalt may also be obtained as bottoms from a VPS in which lubricating oil fractions are being separated. A VPS for the manufacture of asphalts looks very much like the bottom section of a lube VPS. It consists of a tower operated under vacuum in which the reduced crude is flashed. The section below the flash zone of an asphalt VPS is fitted with about six trays to strip with steam the liquid product from the flash zone. This stripping removes lower boiling components present in the liquid, thereby permitting a lower flash zone temperature for the desired cut point. The section above the VPS flash zone is generally fitted with a Glitsch grid. In this section vapors from the flash zone (i.e., VGO product) are condensed by direct heat exchange with a cold VGO liquid pumparound. Liquid from the Glitsch grid is removed from the tower using a total drawoff tray. No fractionation of the vapors from the flash zone is required since there are usually no specifications on the VGO

product. The section above the flash zone in a lube VPS is different than the section above the flash zone in an asphalt VPS. Vacuum distillation minimizes the high temperature exposure of the asphaltic material and thus avoids thermal degradation (i.e., cracking). This is accomplished by use of vacuum which reduces the hydrocarbon partial pressure, making possible the removal of high boiling oil fractions from the asphalt at temperatures well below their normal boiling points. Steam can be injected into the tubes of the VPS furnace to further lower the hydrocarbon partial pressure. This reduces the temperature required for a given degree of vaporization. The steam also prevents thermal cracking by reducing the residence time of the reduced crude in the furnace tubes.

Solvent separation, using the propane deasphalting process, is another procedure by which asphalts of the straight reduced type may be manufactured. This is a physical separation process used to recover high viscosity lube fractions from a given vacuum residuum. When mixed with the residuum, the solvent preferentially dissolves the oil and precipitates the asphalt.

Since the propane deasphalting operation is primarily directed toward the manufacture of lubes with asphalt as a by-product, the grades of asphalt produced from a deasphalter are usually limited. In some cases considerable blending must be done (frequently with extract oils) to provide the variety of grades required by the consumer.

Polymers

Plastics are long chain, high molecular weight polymers which can be formed under application of heat and pressure into desired shapes and contours by a variety of processing techniques. Plastic resins are classified either as thermoplastics, if the material can be reprocessed under heat and pressure, or as thermosets if the material, once formed, cannot be reprocessed.

The plastics industry was launched nearly 150 years ago with the production of certain derivatives of cellulose. However, its real emergence as a growth industry of immense proportions and importance has occurred since World War II with the sustained strong growth of thermoplastics and specialty elastomers. The importance of the polymers industry in modern society is underscored by the myriad of uses already developed for these materials, with more being developed almost daily. Although there are a multitude of polymers that are derived from hydrocarbons, we will only briefly discuss the more common thermoplastics that are made from relatively simple monomers from petroleum.

Polyethylene is the simplest of so-called high polymers. The reaction for low density polyethylene (LDPE) follows the classical free radical polymerization steps of initiator decomposition, initiation, propagation, and termination. The reaction is

triggered by free radical initiators such as organic peroxides or oxygen. These materials decompose rapidly as temperature is increased to form two free radicals:

$$I \rightarrow 2R\bullet$$

These free radicals combine rapidly with ethylene to initiate growing chains:

$$R\bullet + CH_2 = CH_2 \rightarrow RCH_2 - CH_2\bullet$$
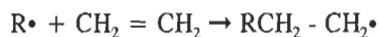
After the polymer chain is initiated, it grows rapidly by successive addition of ethylene monomer molecules:

$$R - CH_2\bullet + CH_2 = CH_2 \rightarrow R - CH_2CH_2 - CH_2 \bullet$$
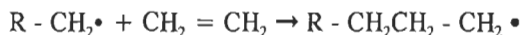
Growing chains are terminated or stop growing by combination or the joining together of two growing chains to produce an inactive polymer molecule:

$$M_n\bullet + M_m\bullet \rightarrow M_{n+m}$$
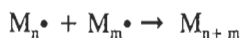
Molecular weight is an important polymer property and is regulated by the addition of small amounts of hydrocarbons called modifiers to the reaction medium. Propylene, butylene, or hexane is used for this purpose. These combine with a growing chain through a chain transfer reaction:

$$M_n \bullet + CH_3CHCH_2 \rightarrow M_n + CH_3CCH_2$$
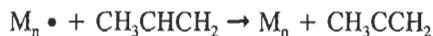
The point of growth is transferred from the polymer chain, which is terminated, to the modifier, which then continues to propagate. Thus, the average length of a polymer chain can be regulated. A reaction which makes the properties of low density polyethylene significantly different from those of high density polyethylene is the short chain branching or "backbiting" reaction:

$$M_n - (CH_2)_4 - CH_2 \bullet \rightarrow M_n - CH - (CH_2)_3 - CH_3$$
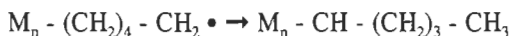
The growing chain bends backward and the point of growth is transferred from the chain end to a carbon atom two to five carbon atoms from the chain end. The chain then continues to grow from this point leaving a short branch. These short chain branches effect polymer properties. By reducing crystallinity, these short branches produce a resin of lower density which is much more flexible than its linear, highly crystalline, high density counterpart. Long chain branching is another reaction affecting LDPE properties. A growing chain can transfer its growth center to another chain where propagation continues producing a long chain which may be as long or longer than the original chain.

Very high purity ethylene is required as feed to facilities making LDPE. Olefin impurities act as modifiers and affect polymer properties; diolefins and acetylenes form gels by crosslinking; paraffinic impurities act as modifiers and diluents affecting reaction control and polymer properties; carbon dioxide acts as a diluent, and is made in the reactor; hydrogen is a modifier; carbon monoxide adversely affects electrical properties for wire and cable coating grades; oxygen is a catalyst synergistic with peroxides, and tends to override the control system if present in concentrations greater than 1 ppm. Sulfur adversely affects product color and odor, and methanol acts as a modifier affecting product properties. Water makes ethylene hydrates and can plug process equipment and reactors. In a typical manufacturing plant, the feed is highly purified ethylene. The feed ethylene is usually compressed in two steps. Primary compressors bring ethylene up to an intermediate pressure of about 4,500 psi. Secondary compressors then compress ethylene to reaction pressure which varies from 25,000 to 50,000 psi depending on the grade being produced. Polymerization modifiers are added to the ethylene feed at the suction to the secondary compressor with intensifier type pumps. Reaction initiators are added directly into the reactor. These are fed into the ethylene stream in a solution high pressure pumps.

The polymerization is carried out at pressures ranging from 25000 psi to 50,000 psi depending on the product being de Two basic types of reactors are tubular and autoclave. A tubular reactor is a long jacketed tube in which initiator can be injected into the ethylene at various points along the reactor, and the heat of reaction is removed by injection of cold ethylene at several points along the reactor, as well as by a cooling fluid in the jacket, The autoclave reactor is typically an agitated vessel. The heat of reaction in the autoclavers is absorbed as sensible heat by the chilled ethylene feed stream. Upon leaving the reactors, the unreacted ethylene, solvents and modifier, are separated from the polymer in two stages. The first stage of the unreacted gas is removed from the polymer by flashing at intermediate pressure. This intermediate pressure ethylene flash gas is chilled and filtered to remove waxes and oils. The recycle gas is normally recycled to the

secondary compressor suction. The reactor product is passed from the intermediate pressure flash drum to a low pressure flash drum. Most of the unreacted ethylene, solvents and modifiers are flashed out of the polymer. The low pressure ethylene is sent to a purge gas purification unit (PGU) for repurification prior to recycle. Recycled low pressure ethylene is added to the primary compressor suction. Molten polymer from the low pressure separator is fed to a hot mail extruder, where it is pelletized, cooled and transferred to bulk storage bins. Additives and stabilizers can be added to the polymer in the hot melt extruder. Figure 30 provides a simplified flow sheet for a LDPE plant, and Figure 31 illustrates ethylene compression and reaction plant.
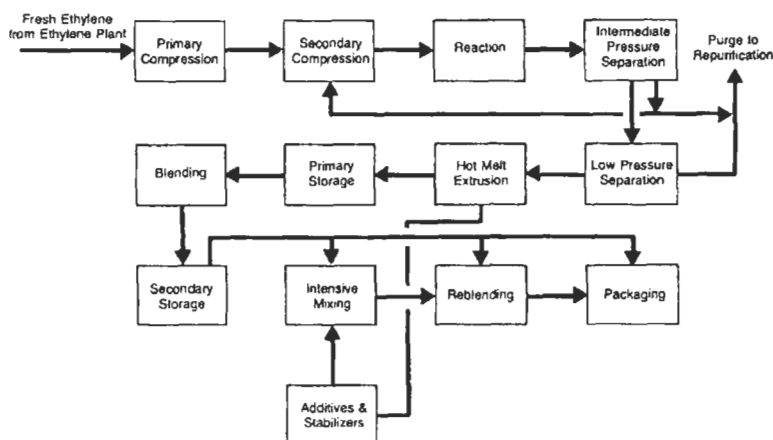
Figure 30. *Example of a LDPE plant.*
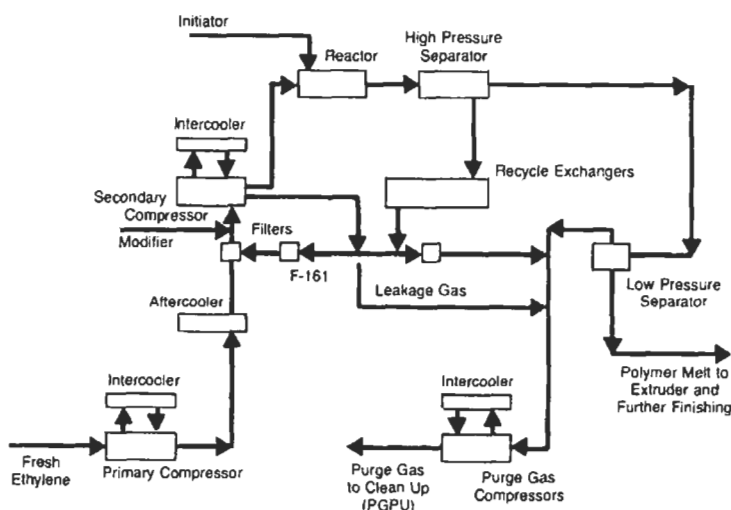
Figure 31. *Example of ethylene compression and reaction plant.*

The next major commodity plastic worth discussing is polypropylene. Polypropylene is a thermoplastic, crystalline resin. Its production technology is based on Ziegler's discovery in 1953 of metal alkyl-transition metal halide olefin polymerization catalysts. These are heterogeneous coordination systems that produce resin by stereo specific polymerization of propylene. Stereoregular polymers characteristically have monomeric units arranged in orderly periodic steric configuration.

The commercially important forms of PP consist of molecules produced in a spiral configuration with pendant methyl groups appearing at regular intervals along one side of the helix. High crystallinity results from this type of structure. Polypropylene exhibits a high yield strength and stiffness, good surface hardness, and an extraordinary flex life. It is impervious to most chemicals and does not undergo environmental stress cracking. These properties coupled with its low density have created a substantial market for PP in molding applications, with particular success in the automotive and appliance industries.

Filaments and fibers are among the largest application areas. High tenacity fibers can be produced by drawing melt spun strands. The molecular orientation which occurs during the drawing process creates a high degree of crystallinity in the polymer, thus giving great strength. Typical applications for these materials include carpet backing, upholstery fabrics, ropes and twine, film and sheeting, and packaging applications. Polymers produced from vinyl-type monomers are essentially linear chain molecules. Polypropylene is a polymer that has steric isomers and is thus known as a stereoregular polymer. There are two orderly fashions in which it can be produced: isotactic, a configurational sequence in which all pendant groups are attached to the same side of the polymer backbone; and syndiotactic, in which the pendant groups alternate from side to side of the chain. These are illustrated schematically in Figure 32.
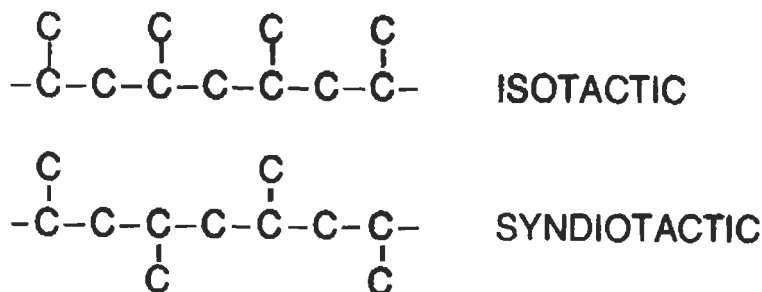
Figure 32. *Configurational sequence of polypropylene.*

A third form, atactic, consists of polymer molecules with a random spatial arrangement of pendant groups. The conformation of the chain is helical, rather than linear. Only the isomeric forms which display sequential regularity are capable of crystallizing into a desirable form with good strength characteristics. Atactic PP is amorphous and rubbery. Of the two orderly, crystalline forms, only isotactic PP is produced on a commercial scale. The form produced is determined primarily by the catalyst employed. The effective catalyst systems from PP consist of various transition metal halides in conjunction with an organometallic compound. These are commonly specific crystalline forms of TiCl, with an aluminum alkyl. Stereoregulation of the propylene polymerization involves controlling the spatial orientation of the monomer as it is added through the propagation step. The entering monomer must be oriented and directed prior to incorporation into the growing polymer chain. This is accomplished by adsorption or complexing of the propylene on the Ti-Al catalyst surface. A commercial PP polymerization catalyst must have several characteristics. It must be highly stereospecific so that a high fraction of the desired molecular isomer is produced. In addition, kinetic rates must be sufficiently high so that catalyst requirements are kept to an economic level. Catalyst is used on a once-through basis with the polymer produced on the surface of the catalyst particles. Process technology is considered proprietary by individual producers and, in general, process details are closely guarded secrets. All the early producers developed their own process and product technologies. The general scheme involves taking liquid propylene and polymerizing it continuously in the presence of an inert xylene diluent. The reactor product is contacted with a diluent like methanol to quench the reaction, precipitate isotactic material that has dissolved in the xylene, and solubilize the polymerization catalyst. The polymer product is recovered from the dissolved catalyst residues and small amounts of undesirable soluble polymer in a filtration step. Diluent and methanol remaining with the polymer are removed in a "water finishing" operation, a combination of water contacting and steam distillation. Polymer is then dried and conveyed to the finishing section. Extensive separation and purification equipment is required to separate diluent, alcohol, and water. The reactor diluent must be absolutely free of methanol and water, which are reaction poisons; the methanol must be dry, as water inhibits solubilization of certain catalyst residues. More modern processes use bulk or gas phase technology, which is beyond the subject matter of this volume.

The last material we will briefly review is polyvinyl chloride (PVC). The term PVC also refers to copolymers of vinyl chloride with other monomers such as vinyl acetate.Some of the advantages of PVC include low cost, the ability to be compounded - resulting in a wide range of flexible and rigid forms, good physical and chemical properties, good outdoor weathering properties (unplasticized), versatility in a broad range of applications, processing ease through a variety of plastics fabricating machinery, self-extinguishing characteristics, and the ability to exhibit good color properties through a wide range of color compounds.

Plasticizers, lubricants, fillers, and stabilizers are used to produce the versatility necessary to meet a wide range of applications. PVC, when modified by plasticizers, produces flexible materials which are desirable for certain applications. Elimination of plasticizers results in compounds termed rigid PVC, that are used in other applications. Flexible PVC is generally used for raincoats, baby pants, dolls, shoes, draperies, garden hose, gaskets, shower curtains, show welting, cable and wire insulation, floor covering, coating on hazardous materials handling garments, etc. The principle markets for rigid PVC are pipe, pipe fittings, conduit, ducting, rain gutters and drain spouts, sliding window tracks and other profile extrusions, house siding, decorative trim and edging, bottles, etc. The conventional approach to manufacturing PVC is via suspension polymerization. Monomer droplets are dispersed in water and polymerized using a monomer soluble, free radical initiator. The particle size is controlled in the 100-150 micron range, and the particle porosity is controlled in the 0-50% range by agitation design and by the use of suspending agents which are colloids and wetting agents. The process is an economical one, and residual concentrations of suspending agent, etc. are low, resulting in good electrical properties and chemical resistance. This is the most versatile process, as the particle properties can be tailored to give optimum processing performance with any of a number of plasticizer levels, and types of processing equipment. Other common manufacturing processes for PVC are bulk or mass polymerization and solution polymerization.

SPIRITS PRODUCTION

An important application of distillation is the production of alcohol and alcoholic beverages. At atmospheric pressure the boiling points of water and ethyl alcohol are 100 °C and 78.3 °C, respectively. Because of its lower boiling point ethyl alcohol is distilled off from a beverage such as wine or beer and enriched in the condensate (distillate) while leaving the water and alcohol at lower concentration behind. It's interesting to note that the water/ethyl alcohol mixture forms an azeotrope. At atmospheric pressure the azeotropic point occurs at a mole fraction of 89.4 % of ethyl alcohol (corresponding to a distillate composition of 95.6 % by weight). By means of simple distillation ethyl alcohol cannot be purified beyond that point. Both brandy and whiskey are distilled beverages. What distinguishes them from each other is the liquor from which they are distilled. Brandy is a distillate of wine, specifically grape wine, while whiskey is distilled from beer and other grain beverages. It is interesting to note that the term used to denote an alcohol distillate, regardless of the language almost invariably translates into the phrase water of life. In Latin it is known as aquae vitae. The French call it eau-de-vie, a term still used to denote French brandies. The English word brandy appears simply means 'burnt wine,' referring to the heating of wine for distillation. Distilled beverages may date

as far back as 800 BC in China with a distillate of Sake. In Europe, distillation was known by at least the eleventh or twelfth century. The Norman English found distillation from grain firmly established when they invaded Ireland in the twelfth century. The Romans found apples in abundance as they made their conquests through Gaul, and as early as the ninth century had laws regulating the production of cider. The first written mention of an eau-de-vie made of 'cyder', however, was not until 1553 when it was mentioned in the journal of a Norman farmer. Arnold de Vila Nova, a 13th Century alchemist, wrote of aqua vitae and its restorative properties and also of the medicinal properties of various flavored alcohols. Legal documents dating to 1411 mention the distillation of wine into brandy in the Armagnac region of France. Distilled alcoholic beverages are possible due to the different boiling points of the two primary ingredients in the original mixture i.e. water and ethyl alcohol. Water boils at a temperature of 100 °C (212 °F), while ethyl alcohol will boil at only 78.3 °C (73 °F). This differential makes it possible to boil out the alcohol from a beverage such as wine, mead, or beer while leaving the water and other substances behind. This is done by heating the liquid to a temperature above 78.3 °C, but below 100 °C. The alcohol, on boiling, is captured and recondensed into a liquid of considerably higher alcoholic concentrations.

The lower the temperature is kept, while remaining in the proper range, the higher the concentration of alcohol will be in the distillate. A lower temperature, and therefore a purer distillate may be desired if the purpose is simply to obtain alcohol to use in fortifying other beverages. In most cases, unless a modern patent still is used, a second or third distillation may be necessary to get a really pure and tasteless distillate. If it is desired, however, to obtain a directly drinkable beverage, a higher temperature will produce a liqueur which contains, in addition to ethyl alcohol, a distinct percentage of water as well as higher alcohols and esters (which are present in the original beverage), keeping the alcoholic content to within range of what can be safely drunk. The inclusion of these other substances will give the beverage flavor and aroma, as well as smoothness of character. These qualities would be lacking in a solution of almost pure alcohol.

Another method, known as fractional crystalization is done by inverting the process and freezing the beverage instead of boiling it. This works for very similar reasons to that of normal heat distillation, namely, the differential in freezing points of the two liquids involved. Water freezes at a temperature of 0 °C, while ethyl alcohol does not freeze until reaching -114 °C. This allows the water to be frozen out of the liquid, leaving behind the ethyl alcohol, as well as the other alcohols and esters. This produces a drink of a rather different character from heat distillation, as it contains everything except water, while heat distilled beverages leave everything behind except alcohol. Note also that simply lowering the temperature to 0 °C will not produce an increase in alcoholic strength. The temperature required for this process is in the range of -15 °C and below, but must vary, much as the diurnal cycle naturally does. This allows crystals of ice to form as the temperature drops.

As the temperature rises slightly the alcohol will drain out of the crystals so that when the temperature again goes down and more crystals of ice re-form they are purer crystals of water, containing less alcohol. As this process repeats itself the solution will gradually work its way toward the alcohol concentrations listed in Table 7.

Table 7. *Alcohol content as a function of Fractional Crystallization Temperature.*

Temperature		Percent Alcohol Content
°F	°C	
10	-12.2	8
5	-15.0	11
0	-17.8	14
10	-12.2	8
-5	-20.6	17
-10	-23.3	20
-15	-26.1	24
-20	-28.9	27
-25	-31.7	30
-30	-34.4	33

Spirits manufacturing is a sophisticated technology, but in general process operations are less complex and demanding as in the case of oil refinery operations. Often the technology may be described as being closer to an art as opposed to an exact science, but clearly engineering principles for distillation, filtration and batchwise type operations are meticulously employed.

CLOSURE AND RECOMMENDED WEB SITES

This chapter has only provided a brief overview of the technology of distillation. In part, to highlight some important engineering concepts and equipment, the following summary is given. As described, refinery main fractionators are distillation towers that separate very wide boiling fractions into a series of rough

product cuts. Usually, the cuts produced are not final products. They go on to further processing and separation. Main fractionators include: atmospheric crude towers, vacuum crude towers, FCC (fluid catalytic cracking) main fractionators, coker main fractionators, visbreaker main fractionators, hydrocracker back end atmospheric and vacuum towers. Other main fractionators less commonly encountered are gas oil towers, vacuum pre-flash towers and atmospheric pre-flash towers (but not preflash drums). Two types of product separation occur in main fractionators. These are (1) separation in the main column and (2) sidestripping in attached sidestripper columns. Sidestrippers come in two varieties; reboiled sidestrippers and partial pressure reduction sidestrippers. The most common type of refinery sidestripper is the partial pressure reduction sidestripper. The motive vapor for this column is usually steam. Natural gas, however, or other superheated hydrocarbons and inerts such as nitrogen are also used in some refineries.

Refinery product separation falls into a number of common classes; namely: Main fractionators; gas plants; classical distillation, extraction (liquid-liquid), precipitation (solvent deasphalting), solid facilitated (Parex(TM), PSA), and Membrane (PRSIM(TM)). This list has been ordered from most common to least common. Main fractionators are required in every refinery. Nearly every refinery has some type of gas plant. Most refineries have classical distillation columns. Liquid-liquid extraction is in a few places. Precipitation, solid facilitated and membrane separations are used in specific applications.

Product separation for main fractionators is also often called black oil separation. Main fractionators are typically used for such operations as preflash separation, atmospheric crude, gas oil crude, vacuum preflash crude, vacuum crude, visbreaking, coking, and fluid catalytic cracking. In all these services the object is to recover clean, boiling range components from a 'black' multicomponent mixture. But main fractionators are also used in hydrocracker downstream processing. This operation has a clean feed. Nevertheless, whenever you hear the term black oil, understand that what is really meant is main fractionator processing.

Gas plants are integrated tower systems intended to recover LPG range material and separate it from naphtha products. This stabilizes the naphtha and reduces its vapor pressure. The LPG material may either be saturate gases going to LPG or unsaturates going to further processing. Gas plants on preflash and atmospheric crude processing units are saturate gas plants. Gas plants on FCC units are unsaturate gas plants. Coker and visbreaker gas plants are somewhere between the two.

Extraction (discussed in Chapter 5) uses the selective adsorption of a component in a liquid to separate specific molecules from a stream. In application extraction may be coupled with its cousins, extractive distillation and azeotropic distillation, to improve extraction efficiency. Typical refinery extraction applications involve aromatics recovery (UDEX) and lubricants processing (furfural, NMP). Extractive distillation and azeotropic distillation are rarely employed in a refinery. The only

standard example is extractive distillation in a Sulfolane(TM) unit. A standard crude processing sequence basically consists of the following. First a preflash is done. A preflash is a drum or tower upstream of the main atmospheric crude column in a crude processing unit. The main sequence is preflash (optional), atmospheric crude, gas oil tower (optional), vacuum preflash tower (optional), vacuum crude (usual). Some plants do not have a vacuum crude tower. Typically, refineries without vacuum units are called topping plants. Many plants have three of the services. The most common combination is preflash-atmospheric-vacuum. Gas oil towers and vacuum preflash towers are much rarer. Normally, plants are built without preflash towers. A new plant of any given recovery and capacity is cheaper to build without a preflash tower than with a preflash tower. Preflash towers are added for two major reasons: first, energy efficiency, second, plant capacity. The energy efficiency benefits of a preflash tower are somewhat dubious. Preflash towers added to a plant are a trade-off between product recovery at a given temperature limit (in general, preflash tower sequences have lower yield recovery for a fixed outlet temperature measured at the atmospheric tower feed point) and higher capacity through the unit. The capacity benefits of preflash are clear and straightforward. The energy recovery aspects of the preflash are more complex. Crude units use heat integration between the downstream towers to recover heat into the feed. The feed then goes to a fired heater to complete the final required heat input of the crude oil. Pressure drop occurs through the preheat train.

There are many excellent textbooks on the subject of distillation, however with the explosion of information on the World Wide Web, process engineers can now gain almost instant access to calculation methods and model simulations which will walk the engineer through the important design steps. The following Web sites are recommended to be reviewed and accessed by the reader.

- **Andrew Sloley's Distillation and Petroleum Refining Technology**: Industrial distillation and refining technology resources as well as links to related sites. *http://@sloley.home.mindspring.com*.
- **Distillation Basics**: About.com article about distillation by Guide Bernhard Spang. *http://C@hemengineer.about.comleducationlchomengineerlli*.
- **Introduction to Distillation**: A distillation tutorial from Ming T. Tham, of the University of Newcastle upon Tyne, United Kingdom *http://Illorien.ncl.ac.uklmingldistilldistilO.htm*.
- **Light Hydrocarbons Separation Technologies**: Site covers proprietary, non-cryogenic, absorption based technology for separation of light hydrocarbons in natural gas, refining and petrochemicals industries. *httij.llwww.aet.com*.
- **Step by Step: the McCabe-Thiele Method**: Introduction to the McCabe-Thiele method for countercurrent multistage distillation, from your About.com Guide, Dr. Bernhard Spang. *http://Chemengineer. about comllibrarylweeklylaa 11 1599.h*.

Chapter 5
MASS SEPARATION EQUIPMENT

INTRODUCTION

Absorption is defined as the process of taking of molecules of one substance directly into another substance. Absorption may be either a physical or a chemical process. Physical absorption depends on the solubility of the substance absorbed, and chemical absorption involves chemical reactions between the absorbed substance and the absorbing medium. Adsorption is a process involving the adhesion of molecules to the surfaces of solids, as opposed to absorption, in which the molecules actually enter the absorbing medium. Charcoal, for example, which has a great surface area because of its porous nature, can adsorb large volumes of gases, including most of the poisonous ones, and thus is used in gas masks and filters. Solvent extraction is defined as a process aimed at the separation of the components of a solution by treatment with an immiscible solvent in which one or more of the components of the solution are soluble. Membrane technology involves the spontaneous passage of a liquid from a dilute to a more concentrated solution across an ideal semipermeable membrane that allows passage of the solvent but not the dissolved solids (solutes). These four technologies or unit operations are aimed at mass separation, relying on chemical properties and concentration driving forces which drive the economics of each process. What we mean by this statement is explained by way of an example. In many situations, for example, distillation is a cost effective means for the separation of components, but it is also true that solvent extraction is sometimes practicable where ordinary distillation is uneconomical or actually impossible. Acetic acid can be recovered from a dilute solution of acetic acid in water by distillation or by extraction with a solvent like toluene, in which the acid is soluble and the water is insoluble. In contrast, close-boiling petroleum fractions, differing chemically, may be almost impossible to separate by distillation but are easily separable by means of solvent extraction.

This chapter provides a very elementary overview of the engineering basics of four important mass separation techniques. Fundamental concepts are stressed rather than actual equipment, largely because equipment and applications are often specific as opposed to off-the-shelf or standardized designs. The techniques are based on widely different separation techniques than those that rely on physical means, such as filtration, settling, centrifugation, and others. Physical separation technologies and equipment are discussed in Chapter 6. The subject matter treated presently is

well established, and indeed modern day engineering principles, particularly absorption and extraction, were established nearly 70 years ago for applications in the petroleum and chemical processing industries. The fundamental engineering concepts developed in the 1930's through early 1950's are still quite applicable to today's designs and continued to be used.

ABSORPTION EQUIPMENT

Absorption is an old but still widely used technique that is used in the selective removal of a gaseous contaminant or product from a gas mixture. The constituent may be removed because of its economic value, or as a stage in the preparation of some compound, or because it is an air pollution problem. Removal is often effected by absorption in a liquid in which only the gas concerned is soluble. It is sometimes also necessary to free a liquid from dissolved gases. This can be achieved by contact with an insoluble gas, in which case the process is known as stripping (the reverse of absorption). Common examples of these processes are found in the absorption of sulphur trioxide in the manufacture of sulphuric acid and oleum, the scrubbing of plant exit gases to free them from hydrogen sulphide or sulphur dioxide before discharging them into the atmosphere, and the stripping of carbon dioxide from solution in water.

The objective of absorption equipment is to provide intimate contact between the liquid concerned and gas, thus facilitating the absorption process. When a large volume of gas is absorbed per unit volume of liquid, a marked rise in temperature may result and devices for the removal of the heat evolved are then an important feature of the equipment. Apart from packed towers, the types of equipment described here include agitated vessels, bubble-plate towers, cooler-absorbers and spray towers.

Agitated Vessels: These are vessels in which the gas is introduced directly into the liquid and is mechanically dispersed by means of a suitable agitator. They are used mainly where the absorption is accompanied by a slow chemical reaction between the dissolved gas and some constituent of the liquid, and sufficient time has to be allowed for this reaction to proceed to the required extent. Alternatively, they may be used where close control of the process is required in order to prevent the formation of undesirable by-products. Agitated vessels are suitable for batch operations. If used for a continuous process, counter-current flow cannot be obtained within individual vessels, and it is then almost always necessary to use a series of vessels arranged in multiple stages. The pressure-drop through agitated vessels is high compared with that for packed towers, and where this is an important factor, the use of a series of packed towers, through which a proportion of the liquid is circulated repeatedly, might be preferable.

Bubble-Plate Towers: These towers resemble the classical bubble-cap columns used for distillation. They consist of a number of shallow plates or trays over each of which the liquid flows in turn on its way down the tower. The gas enters at the bottom of the tower and is made to flow through a number of bubble caps on each plate. These caps may be of various shapes, though they usually take the form of inverted cups, and their edges are slotted so that the gas escapes from them into the liquid in the form of bubbles. The layout of a typical plate is illustrated Figure 4 of Chapter 4 (page 168). The illustration shows the arrangement of bubble caps on a plate together with the risers through which the gas enters the bubble caps and the downcomers which carry the liquid from plate to plate. The inlet weir assists in distributing the liquid over the plate, while the outlet weir maintains the desired depth of liquid. Bubble-plate towers may be preferred to packed towers when:(a) the liquid rate is so low that a packed tower could not be used effectively since the packing would not be adequately wetted; (b) when a difficult absorption duty is required, i.e. the composition of the gas at either end of the tower approaches that corresponding to equilibrium with the liquid. While faulty gas and liquid distribution can generally be tolerated in a packed tower, they may so much reduce its efficiency under these conditions as to prevent the desired performance from being achieved; (c) there is a risk that solid matter may be deposited. Bubble-plate towers, which can be fitted with manholes, are more easily cleaned than packed tower configurations; (d) a slow chemical reaction occurs in the liquid. As with agitated vessels, the use of a bubble-plate tower allows time for the chemical reaction to proceed to the desired extent.

The pressure-drop through bubble-plate towers is high compared with that for packed towers and is of the order of 1 to 2 in. of water per plate. For difficult absorption duties where the pressure-drop available is limited, the use of a combination of packing and bubble plates may lie considered- In this arrangement a few plates are installed at the end of the tower where the composition of the gas approaches that corresponding to equilibrium with the liquid. The performance of bubble-plate towers is expressed in terms of plate efficiencies. These indicate the extent to which equilibrium between gas and liquid is attained on the plate. Though high efficiencies have been achieved with bubble-plate columns in distillation, it is important to realize that plate efficiencies in absorption or stripping may be less than 10% if the gas concerned is of low solubility.

Cooler Absorbers: When the absorption of a gas is accompanied by the evolution of heat, an important function of the absorption equipment is the removal of the heat generated. This may be accomplished by using a number of towers in series, the liquid from each tower being circulated through an external cooler. There are different types of cooler-absorbers in which processes of this type can be carried out in a single unit. The materials of which these cooler-absorbers are constructed should be of high thermal conductivity and resistant to corrosion by the substances used in the process. As an example, in the manufacture of hydrochloric acid of the

highest possible concentration, for which purpose the liquid must be cooled as absorption proceeds, the materials used include glass, tantalum and impervious carbon. Glass condenser units, consisting of a number of layers of pancake glass coil joined together in series and fitted into a length of standard glass pipe, have been used in this process. Another type of cooler-absorber also used in the production of hydrochloric acid is made of impervious carbon and is fitted with transverse tubes carrying the cooling medium, with the absorbing liquid flowing downward over the outside of the tubes and the gas flowing upward. Other types, such as stoneware tourills and silica S-bends, once widely used for the same process, are now regarded as obsolete, but may still be found in use in parts of the former Soviet Union today.

Spray Towers: A spray tower consists of an empty shell into the top of which the liquid is sprayed by means of nozzles of various kinds; the droplets thus formed are then allowed to fall to the bottom of the tower through a stream of gas flowing upwards. The use of sprays appears to offer an easy way of greatly increasing the surface area exposed to the gas, but the effectiveness of the method depends on the production of fine droplets. These are difficult to produce and suffer from the disadvantage that they are liable to entrainment by the gas even at low gas velocities. The surface area may also be reduced as a result of the coalescence of the droplets first formed. As a consequence of these effects, the large increase in surface area expected may not be achieved, or if achieved may be accompanied by serious entrainment and internal circulation of the liquid so that true counter-current flow is not obtained. A single spray tower is suitable for easy absorption duties. For difficult duties, a number of towers in series can be used.

Packed Towers (also known as wet-scrubbers): A packed tower is a vessel that is filled with some suitable packing material. The liquid is introduced at the top of the tower and flows down through the packing to the bottom, and in so doing, exposes a large surface area to contact with the gas. The gas may be introduced at either end of the tower since the gas and liquid flows may be either co-current or counter-current. The counter-current operation is most commonly used.

Numerous types of packing have historically been employed ranging from lumps of coke or quartz to complicated manufactured ceramic and plastic shapes. The arrangement of a typical packed tower is illustrated in Figure 1.

The role of the packing material is to provide a large surface area, and must have a geometry that provides large void space when packed in order to allow for good fluid flow characteristics. Raschig rings are a common example, consisting of hollow cylinders having an external diameter equal to the length of the object. Other shapes include Berl saddles, Intalox saddles, Lessing rings, cross- partition rings, spiral-type rings, and drip-point grid tiles. These objects are usually dumped into the column randomly, or if regularly shaped packing is used, stacked in an orderly fashion.
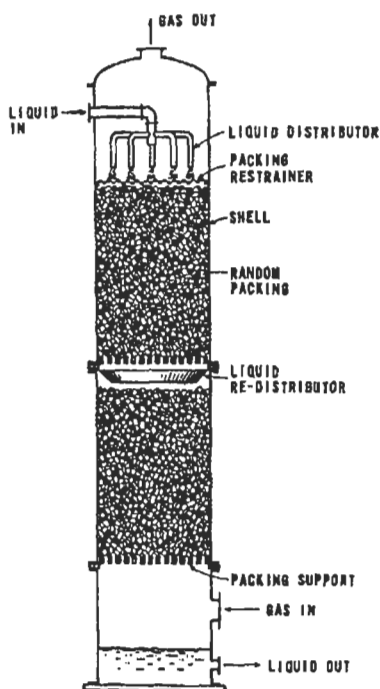
Figure 1. *Packed tower wet scrubber.*

In general, randomly dumped packing has a higher specific surface contact area and a higher gas pressure drop across the bed will be experienced. Stacked packing arrangements have an advantage of lower pressure drop and higher possible liquid throughput, but generally are more expensive to construct. The effectiveness of a packed tower depends on the availability of a large, exposed liquid film. Hence, if poor liquid distribution prevents a portion of the packing from being irrigated, then part of the tower becomes ineffective. Liquid mal-distribution can result from improper introduction of the liquid at the tope of the tower and channeling within the tower. The liquid rate must be sufficient so as to provide sufficient wetting of the packing material, but not so great as to flood the tower. Important terms to note are load point and flood point. These terms are used to define tower capacity. For a given packing and liquid rate, a plot of gas pressure plotted against gas velocity on a logarithmic scale, identifies two break points where the slope of the curve increases. At low gas velocities the curve is nearly parallel to that obtained with dry packing, but above the break points, pressure drop increases more rapidly with increased gas velocity. The lower of these two breaks is known as the load point and the higher one is the flood point. As gas velocity increases above the load point, the liquid holdup in the packed bed increases until, at the second break point (the flood point), most of the void space in the tower is filled with liquid and liquid

entrainment in the gas stream occurs. There is excessive pressure drop during this operation. Columns should seldom be operated above the load point, however in practice towers generally operate between 40 and 70 percent of the flood point. In general, flooding velocities are considerably higher for stacked packing arrangements than for dumped packing.

Venturi Scrubbers: These devices are discussed elsewhere in the volume, but a few comments are worthy within this subject entry. Like spray towers and spray chambers, equipment that relies on the venturi principle are mainly used for removing particulate matter from gas streams, however there are some applications in gas absorption. With gas absorbers based on the venturi principle, the required interface contact is achieved by differences between the gas and liquid droplets, and by the turbulent nature of the venturi throat. Dispersion in a venturi scrubber is achieved by first injecting the liquid into the gas stream as it passes through the venturi, or by admitting the gas to the liquid stream as it passes through the venturi. In the latter scenario, the venturi also serves as a vacuum-producing device and inspirates the gas into the venturi throat. In both arrangements, a gas-liquid separation chamber is used to prevent entrainment. Venturi systems are able to achieve a high degree of liquid-gas mixing, but have the disadvantage of a relatively short contact time which generally leads to poor absorption efficiency. However, for gas systems with high solubilities and proper selection of the scrubbing liquid, the venturi can be an excellent devise. A major disadvantage is the high pressure drop with attendant high poor requirements needed for operation. In all types of equipment used for gas absorption, an essential feature is that the gas and liquid are brought into direct contact. If absorption is then to take place, the soluble constituent of the gas mixture must be transferred from the gas phase to the liquid phase. This is brought about by the diffusion of the soluble gas through the gas phase to the gas/liquid interface and hence into the liquid phase. Absorption is a diffusion process.

The driving force bringing about the diffusion arises from the difference in concentration of the soluble gas in the two phases. Just as the passage of an electric current through a circuit is opposed by the resistance of that circuit, the transfer of material by diffusion is opposed by diffusion resistance. The diffusion of the soluble gas will encounter resistance in both the gas and liquid phases. The so-called "two-film theory" provides a simple picture of the processes taking place which, while probably not corresponding closely to reality, usually provides a satisfactory basis for the design of absorption towers. The theory assumes that when a gas and a liquid are brought into contact, the main bulk of both fluids is in turbulent flow and thus uniformly mixed. Adjacent to the interface, however, and on either side of it, there are assumed to be thin films of gas and liquid in streamline flow. It is further assumed that the resistance to the diffusion of the soluble gas are located wholly in these thin films and that the main bulk of both fluids together with the interface itself offers no resistance. The principle is illustrated in Figure 2.

The rate at which the process of absorption takes place can be expressed by the following relationship:

$$\text{Rate of Absorption} = \text{Absorption Coefficient} \times$$
$$\text{Surface Area} \times \text{Driving Force}$$

Expressions of this type can be written for both gas and liquid films in which the absorption coefficients are the gas- and liquid-film coefficients, respectively. The driving force across the gas film is given by the difference between the actual partial pressure of the soluble gas and that at the interface, while the driving force across the liquid film is given by the difference between the concentration of the soluble gas at the interface and that in the main bulk of liquid.

For an infinitesimal portion of the interfacial area dA at any point in the tower the two equations are:

$$dW = k_g \times dA \times (p_g - p_i)$$
$$dW = k_l \times dA \times (c_i - c_l)$$
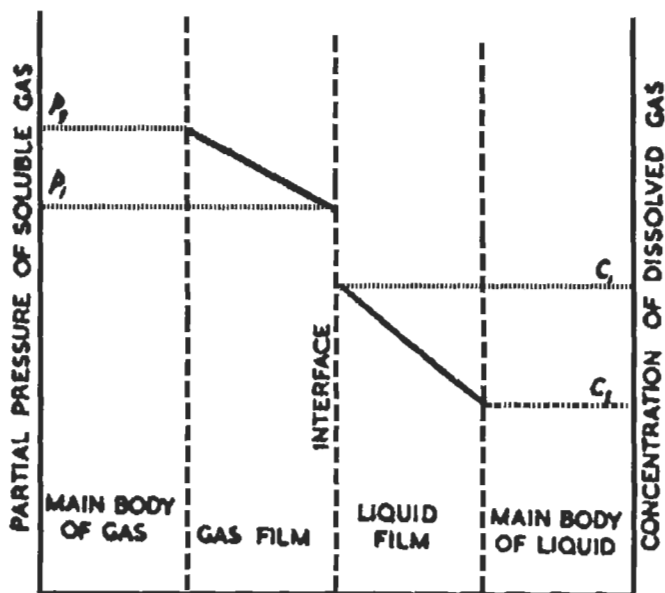
Figure 2. *Two-film theory.*

where: W = rate of absorption

k_g = gas-film coefficient

k_l = liquid-film coefficient

A = interfacial area

p_g = actual partial pressure of soluble gas

p_i = partial pressure of soluble gas at interface

c_i = concentration of soluble gas at interface

c_l = actual concentration of soluble gas in liquid.

The use of these equations is not convenient, since conditions at the interface are difficult to determine. Overall coefficients are therefore introduced by means of which the rate of absorption across both films together may be calculated. These overall coefficients can be defined in the following two ways depending upon the manner in which the overall driving force is expressed: (1) On the gas-phase basis - the overall driving force is then the difference between the actual partial pressure of the soluble gas p_g and the partial pressure p_i corresponding to equilibrium with a liquid of concentration c_1; (2) On the liquid-phase basis - the overall driving force is then the difference between the concentration c_g corresponding to equilibrium with gas of partial pressure p_g and the actual concentration of the soluble gas c_1. Thus, equations for the rate of absorption across both films together can be stated as follows:

$$dW = K_g \times dA \times (p_g - p_1)$$
$$dW = K_l \times dA \times (c_g - c_1)$$

where K_g = overall gas-phase coefficient

K_l = overall liquid-phase coefficient

p_1 = partial pressure of soluble gas in equilibrium with liquid of concentration c_1

c_g = concentration of soluble gas in equilibrium with gas of partial pressure p_g

Overall coefficients cannot be predicted directly from the physical properties and flow rates of a system but must be derived from the individual film coefficients. It is important therefore to be able to relate overall and film coefficients. This can be done if it is realized that the coefficients are in effect conductances. The diffusional resistance of the two films are equal to the reciprocals of the film coefficients, and

by the usual law of the additivity of resistance in series, the sum of these reciprocals is equal to the reciprocal of the overall coefficient. Allowance has to be made, however, in adding the film resistance for the fact that partial pressures and concentrations are not measured in the same units. A factor called the solubility coefficient H is therefore introduced to allow for this difference of units. The equations relating the film and overall coefficients are:

$$1/K_g = 1/k_g + I/Hk_1$$

$$1/K_1 = H/k_g + 1/k_1$$

where H = solubility coefficient, i.e. the ratio of change in concentration of dissolved gas to change in equilibrium partial pressure. According to Henry's law

$$c_1 = \text{Constant} \times p_1$$

For cases where this law holds, the solubility coefficient H will be constant provided that the temperature remains constant, otherwise its value will vary with concentration. The overall coefficient can therefore change as the absorption process proceeds. Packed towers historically have been the most widely used configuration in an absorption tower. The function of tower packing is, basically, to provide intimate contact between the gas or vapor phase and the liquid phase with a minimum resistance to flow through the tower, with the purpose of providing mass transfer between the two phases. Distillation involves the transfer of mass from the liquid to the gas phase, while absorption involves mass transfer from the gas to the liquid phase. Absorption is practiced for two primary reasons, one is to recover valuable products by removal from the gas stream. For example, in petroleum production, natural gasoline is removed from well-head gas systems by absorption into a special hydrocarbon oil. Absorption is also practiced in industrial chemical manufacturing as an important operation in the production of chemical compounds. For example, in the manufacture of hydrochloric acid, one step in the process involves the absorption of hydrogen chloride gas in water. The second basic type of absorption process is the removal of a contaminant from the gas stream allowing purification of the gas or vapor. In air pollution control, this process is often called scrubbing. Among the gaseous air contaminants most commonly controlled by scrubbing are sulfur dioxide, hydrogen sulfide, hydrogen chloride, chlorine, ammonia, oxides of nitrogen and light hydrocarbons. Gas absorption, or scrubbing, is a diffusional process involving the transfer of gas molecules into the liquid phase. Concentration differences between the two phases at the points where they came into intimate contact serves as the driving force. The chemical industry

has utilized packed towers for over 100 years. In the early stages of development, towers were filled with such available materials as broken glass, broken crockery, random threaded nuts, or other non-uniform shaped, inert materials giving a large void space when packed. Rock and gravel were used in some cases, but had the major disadvantages of being very heavy and having small surface area, giving poor performance and fluid flow capacity. Also, they were at times, not chemically inert. Lumps of coke were also used eliminating some of the weight disadvantages. The coke due to its porosity, had large surface area per unit volume. However, the exposed surface area was not completely effective as the pores are so small that they become filmed over by the scrubbing liquid, or solvent, reducing the effective surface area. These packings, all had the disadvantage that performance could not be accurately predicted, and depended on the characteristics of the packing materials obtained in each case. Therefore, no two operations performed the same in early designs.

In 1915, the development of Raschig Rings, by Frederick Raschig, gave packed tower columns a degree of predictability and dependability by providing cylindrical sections, having diameter equal to the length available, allowing varying degrees of packing openness, and flexibility with packings which provide much higher capacity and performance than the original uniformly shaped packing. Raschig Rings have been made to suit various diameters over the years with regard to performance and pressure drop. There are a wide variety of tower Raschig Rings. Tower packings have developed to the point where they now provide serious competition to mass transfer applications previously limited to trays. Usually tower designs are such that the flow is countercurrent, with the liquid introduced at the top of the tower and therefore flows down through the packing, while the gas is introduced at the bottom of the tower and flows upward through the packing material. This gives maximum "driving force" for the transfer of the solute into the solvent throughout the entire column. For this reason, this type of scrubber design is commonly used to provide high-efficiency removal of gaseous contaminants. Also, this design is most effective in removing low solubility gases. From an economic viewpoint this scrubber design generally results in a lower initial capital investment than the other scrubber designs available. However, this type of unit requires higher operating costs due to higher pressure drops and larger liquid flow rates than required in other packed bed designs.

In co-current flows, shown in Figure 3, the gas stream and scrubbing liquid flow in the same direction, generally from the top of the column downward. In this configuration there is initially a very high transfer of the solute into the solvent. This continually decreases during the flow downward through the column due to the continually decreasing concentration difference between the gas and liquid. Co-current scrubber designs may be considered for removing gases which have a high solubility in the scrubbing liquid. However, they are not suitable for removal of contaminants with low solubility because of the low "driving force", once some of

the solute has been dissolved into the solvent. This type of design is considered when there is limited space available for scrubber installation because it can be operated at much higher gas flow rates per unit area and with considerably higher liquid loadings. This is due to the fact that packing flooding is not a consideration since liquid and gas are flowing in the same direction. Co-current flow might be in the case of a very tall column built in two sections, both located on the ground. The second section might be designed for co-current flow as an economy measure to eliminate the need for construction of a large gas transfer pipe from the top of the first column to the bottom of the second. Also, for operations requiring extremely high solvent flow rates, co-current flow might be used to prevent flooding that could occur in counter-current operations. In a cross-flow scrubber design, as shown in Figure (4), the gas flows horizontally through a packed section with the solvent sprayed on the packing from the top. The gas flows perpendicular to the scrubbing liquid. This type of scrubber design is considered when removal of solid, or liquid particulate 5 microns in diameter or larger is required. This is accomplished by impingement of the particulate on the packing surface as the gas flows horizontally through the packing.
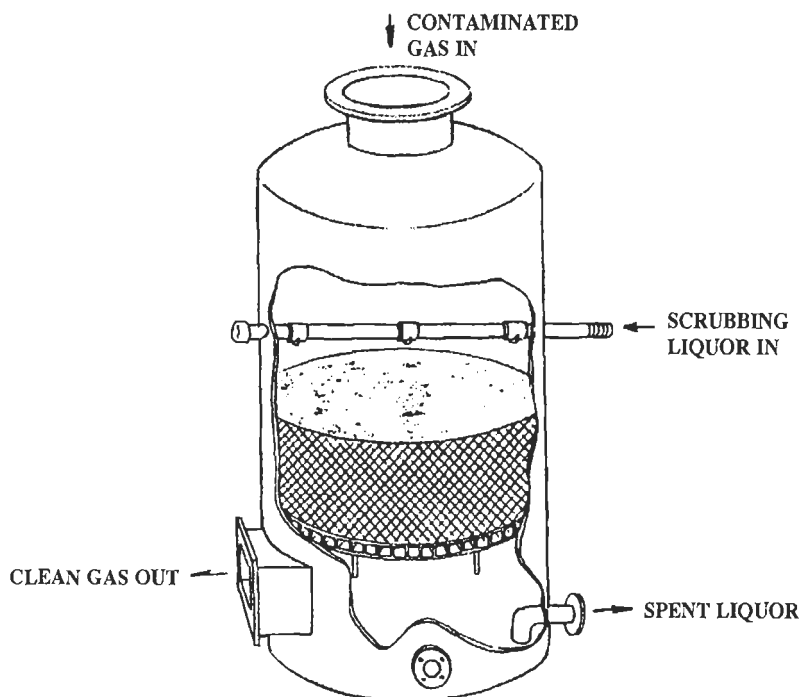
Figure 3. *Co-current scrubber configuration.*
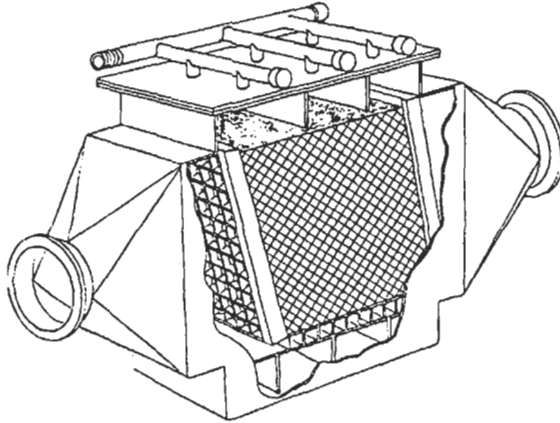
Figure 4. *Example of a cross-flow scrubber.*
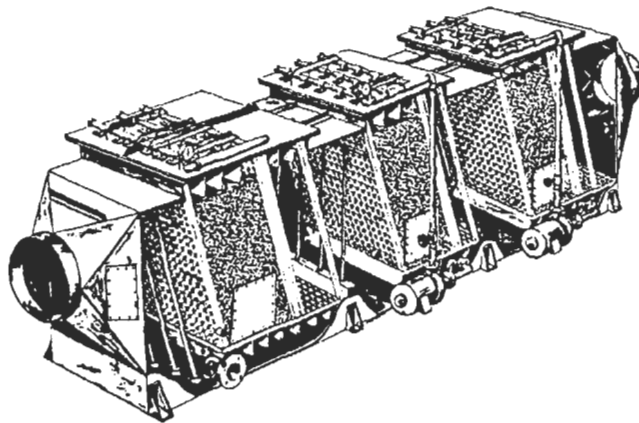
Figure 5. *Example of multiple bed cross-flow scrubber.*

These particles tend to flow in a straight line rather than following the gas stream lines around the surfaces of the packings. Once the particles hit the wet surfaces of the packing, they stick to the surface and are then flushed away by the continuing flow of scrubbing liquid. The cross-flow scrubber design is also considered when gas scrubbing, in addition to the particulate scrubbing, is required. This design offers substantial benefits when removal of several gas contaminants requires the use of several different liquid scrubbing media. By utilizing multiple, separate beds of packing, as shown in Figure 5, each bed can be wetted with a different scrubbing media, thereby preventing the liquids from mixing during the scrubbing operation.

As stated, gas absorption is a process whereby gas and liquids are brought into intimate contact and a component of the gaseous phase becomes dissolved in the liquid. With the proper selection of scrubbing liquid, the gas contaminant will have

a high solubility, allowing maximum transfer of the gas molecules into the scrubbing liquid. At the interface between the gas and the liquid streams, the velocities, in the respective phases, reduce so that a small zone, called the laminar region. It is in this zone, near the gas/liquid interface, where intimate contacting between the gas and liquid phases results. Diffusion from the laminar film of the gas into the laminar film of the liquid, is on a molecular scale, and is called molecular diffusion. A concentration difference, or gradient, between the gas and liquid, is established in the laminar interface region, and it is this concentration gradient which results in gas absorption across the interface. This diffusion does occur from the gas to the liquid and also from the liquid to the gas. A properly designed scrubber, will, of course, have a much higher transfer rate from the gas to the liquid phase. Equilibrium concentration is described as the concentrations of the solute in liquid and gas where there is equal transfer from gas to liquid and liquid to gas. Therefore, no matter how long the gas and liquid remain in contact, there will be no change in concentrations of the solute between the two phases. This rate of diffusion can be determined for gas/gas and liquid/liquid systems in the following manner: in a gas/gas steady state system, molecular diffusion of a component A from position 1 to position 2 through a stagnant non-diffusing component B is determined by:

$$N_A = D_{AB} \, P \, (P_{A1} - P_{A2}) \, / \, RTZ_g P_{BM}$$

The diffusion coefficient can be determined from the Hirschfelder-Bird-Spotz equation as follows:

$$D_{AB} = 0.0009292 T^{3/2}[(1/M_A) + (1/M_B)]^{\frac{1}{2}} \, / \, P\gamma_{AB}^2[\mathscr{F}(kT/\epsilon_{AB})]$$

Parameters in the above expressions are defined as follows:

NA = Rate of diffusion of component A (moles/hr/ft^2)

D_{AB} = Diffusivity (cm^2/sec or ft^2/hr)

R = Universal gas law constant (82.06 cm^3-atm/g-mole or 0.729 ft^3-atm/lb-mole)

P_{BM} = log-mean pressure of non-diffusing component B (atm)

P_A, P_B = Partial pressure of component A and B (atm); subscripts 1,2 indicates interface and equilibrium, respectively

T = Absolute temperature (°K)

Z_g = Distance in direction of diffusion (cm)

γ_{AB} = Molecular separation at collision (Å)

k = Boltzman's constant (1.38×10^{-16} ergs-°K)

ϵ_{AB} = Energy of molecular interaction of A and B (ergs)

M_A, M_B = Molecular weights of components and B, receptively

The collision function, $\mathscr{F}(kT/\epsilon_{AB})$, is given in Figure 6. Values of ϵ/k (in units of °K) and (Å) for common gases are tabulated in Table 1.

The diffusivity coefficient for a liquid-liquid system can be estimated from the Wilke correlation:

$$D_{AB} = T/\mu_B F$$

where μ_B = Viscosity of the solvent (cps)

F = Function of the molecular volume of the solute. Correlations for this parameter are given in Figure 7 as a function of the parameter ϕ, which is an empirical constant that depends on the solvent characteristics. As points of reference: for water, $\phi = 1.0$; for methanol, $\phi = 0.82$; and for benzene, $\phi = 0.70$. The two-film theory is convenient for describing gas-liquid mass transfer where the pollutant solute is considered to be continuously diffusing through the gas and liquid films.
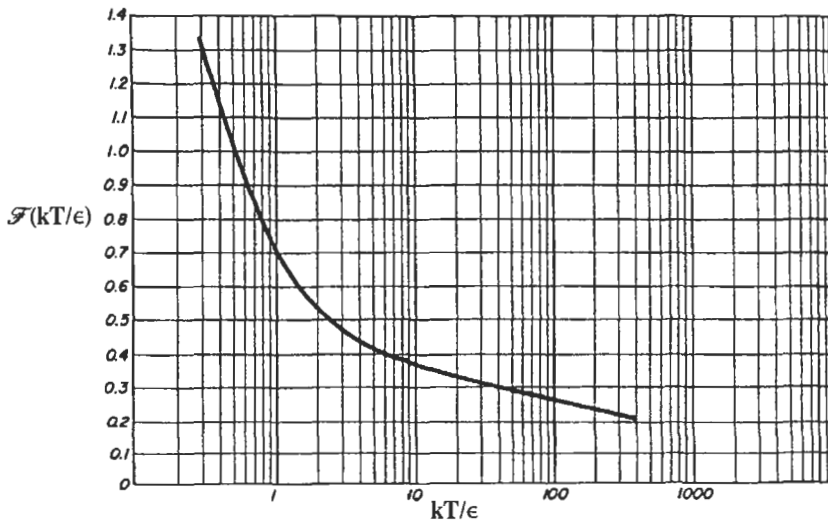
Figure 6. *Plot of the collision function.*

Table 1. *Values of Force Constants and Collision Diameters*

Gas	ϵ/k (°K)	γ_o (Å)
Air	97.0	3.617
Ammonia	315	2.624
Argon	124	3.418
Benzene	440	5.570
Carbon Dioxide	190	3.996
Carbon Monoxide	110.3	3.590
Carbon Tetrachloride	327	5.881
Diphenyl	600	6.223
Ethane	230	4.418
Ethanol	391	4.455
Ethyl Ether	350	5.424
Ethylene	205	4.232
Fluorocarbon F-12	288	5.110
Helium	6.03	2.70
n-Heptadecane	800	7.923
Hydrogen	33.3	2.968
Hydrogen Chloride	360	3.305
Iodine	550	4.982
Methane	136.5	3.882
Neon	35.7	2.80
Nitrobenzene	539	4.931
Nitrous Oxide	119	3.47
Nitrogen	91.5	3.681
Nitrogen Dioxide	220	3.879
n-Octadecane	820	7.963
n-Octane	320	7.451
Oxygen	113.2	3.433
Propane	254	5.061
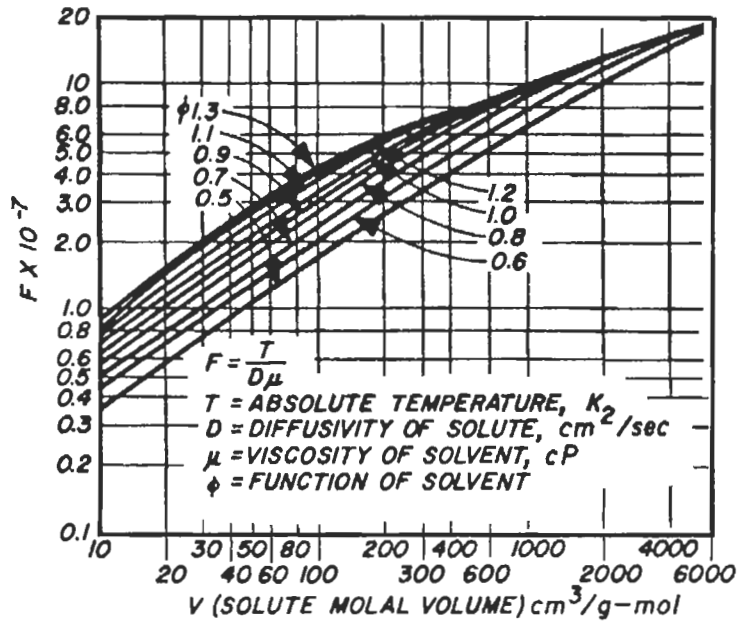
Figure 7. *Solvent characteristic, F, versus solute molal concentration.*

The driving force for the absorption process is the concentration difference between the gas and liquid phases, which in terms of the interface, can be expressed by partial pressures:

$$N_A = k_g (P_A - P_{Ai}) = k_\ell (c_{Ai} - c_A)$$

k_ℓ and k_g are the liquid and gas phase mass transfer coefficients, which can be determined from the following relationships:

$$k_\ell = D_{ABg}P/RTZ_g P_{BM}$$
$$k_g = D_{AB\ell}/Z_\ell$$

Since it is difficult to measure interfacial concentrations of the gas and liquid film (P_{Ai} and c_{Ai}) and the distance in direction of diffusion (Z_g and Z_ℓ), this problem can be eliminated by using the over-all mass transfer coefficients, K_ℓ and K_g, for the liquid and gas respectively. The rate of absorption of the solute depends on the concentration difference or gradient of the solute between the liquid and gas phases. This concentration gradient can also be expressed in terms of the difference between

the bulk concentrations, and the equilibrium concentration described earlier. This equilibrium concentration may be defined by:

$$P_A^* = Hc_A$$

where H is Henry's law constant. When the equilibrium concentration is substituted for the interfacial concentration, then the local mass transfer coefficients can be expressed in terms of the overall mass transfer coefficients as follows:

$$N_A = k_\ell(c_{Ai} - c_A) = K_\ell(c_A^* - c_A)$$
$$= k_g(P_A - P_{Ai}) = K_g(P_A - P_A^*)$$

The relationships between the local and overall mass transfer coefficients are:

$$1/K_g = 1/k_g + H'/k_\ell$$
$$1/K_\ell = 1/k_\ell + 1/(H'k_g)$$

Note that H' is simply Henry's constant corrected for units. When the solute gas is readily soluble in the liquid solvent, Henry's law constant (H or H') is small and K_g approximately equals k_g, and the absorption process is controlled by the gas film resistance. For systems where the solute is relatively insoluble in the liquid, H is large and K_ℓ approximately equals k_ℓ and the absorption rate is controlled by the liquid phase resistance. In most systems, the solute has a high solubility in the solvent selected, resulting in the system being gas film resistance controlled.

For any particular system, a graph can be constructed using the concentration of the solute in the liquid phase (c_A) and the concentration or partial pressure of the solute in the gas phase (P_A) as the abscissa and ordinate, respectively. A line indicating the equilibrium concentrations of the solute in the gas and solvent drawn on this graph, results in an equilibrium diagram.

Generally, these concentrations are expressed in terms of moles of solute per mole of pure solvent (liquid phase) and moles of solute per mole of inert gas (gas phase), thus making the material balance calculations easier.

A typical equilibrium diagram is shown on Figure 8. As noted earlier, a counter-current flow scrubber provides the highest efficiency of operation. The respective concentrations and rates, as shown in Figure 9 provide the basis for a material balance across this scrubber design.
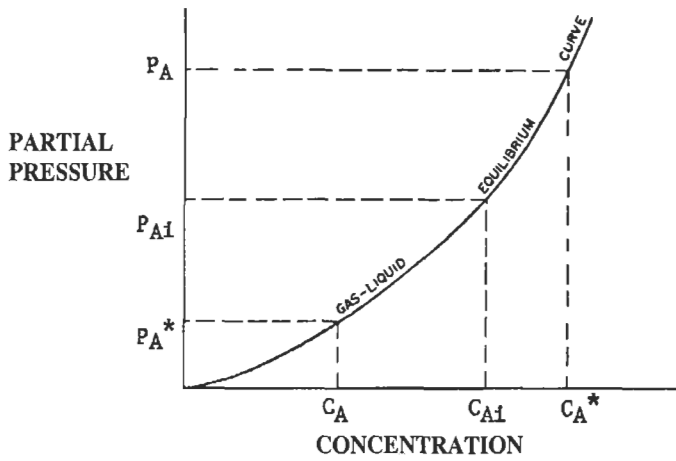
Figure 8. *Gas-liquid equilibrium curve.*
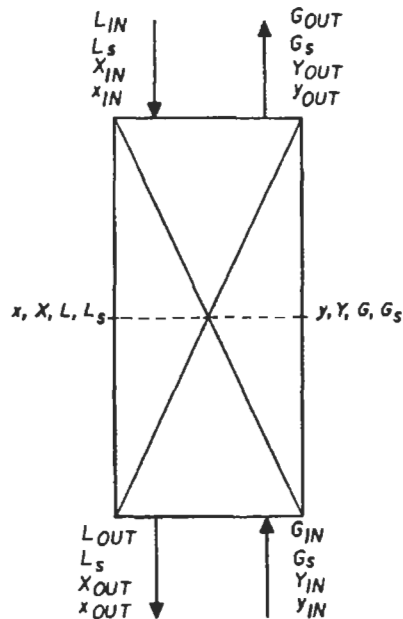
Figure 9. *Material balance diagram.*

Defining the following variables for the purposes of a material balance:

$$X = x/(1 - x); \quad Y = y/(1 - y)$$

From Figure 9, a material balance across the scrubber section shown is as follows; providing a basis for a graphical method of tower sizing:

$$G_s = (Y_{in} - Y_{out}) = L_s(X_{out} - X_{in})$$

$$Y_{in} - Y_{out} = (L_s/G_s)\{X_{out} - X_{in}\}$$

L_s and G_s are the pure liquid and inert gas loading rates, respectively, in units of lb-moles/hr-ft^2. The second expression is the operating line on an equilibrium diagram. In all scrubbing application, where the transfer of solute is from the gas to the liquid, the operating line will lie above the equilibrium curve. When the mass transfer is from the liquid to the gas phase, the operating line will lie below the equilibrium curve. The latter case is known as "stripping".

The development of the operating line is required to determine the minimum liquid flow rate requirement for scrubbing. In an actual scrubber design, all of the following information will be known: the concentration of the solute in the gas (Y_{in}), the concentration of the solute in the entering solvent (X_{in}), the entering gas flow rate (allowing calculation of G_s), and the required concentration of the solute in the exit gas (Y_{out}). To calculate the minimum liquid flow rate (L_s), concentrations Y_{in} and X_{out} will be at equilibrium (on the equilibrium line at the point Y_{in}, X_{eq}). The slope of the operating line between X_{eq}, Y_{in} and X_{in}, Y_{out} represents the condition resulting in minimum required flow rate, as shown on Figure 10.
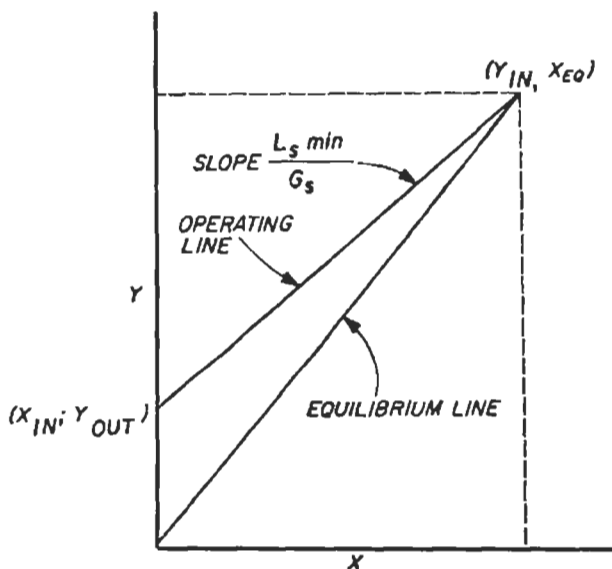
Figure 10. *Operating line for absorption.*

The slope of the line is L_S/G_S. This procedure provides the minimum theoretical solvent requirements. In practice, however, liquid rates are selected at least 25 to 100% greater than the theoretically calculated minimum rate. Typically, packing manufacturers suggest that liquid rates between 2 and 5 gpm/ft^2 result in the best operation. Higher liquid flow rates will effect the degree to which the packing surfaces become fully wetted. Obviously, higher liquid flow rates will result in a greater portion of the packing surface being wetted, thereby giving higher active liquid gas contacting area and a higher rate of mass transfer. When approached from an economic standpoint, higher liquid flow rates results in a higher efficiency of scrubbing per unit tower volume, but lower concentration of the solute in the solvent. This results in a higher quantity of scrubbing liquid used, which generally means a higher operating cost, especially where solvents are valuable. However, this design approach results in minimum initial capital investment. Where lower liquid flow rates are considered, the result is larger scrubbing towers, resulting in higher initial capital investment cost, but lower operating usage of scrubbing liquid. Scrubbing manufacturers advise that above a certain liquid flow rate, known as "minimum wetting rate" (MWR), there is virtually no increase in mass transfer rate. This is due to the fact that at this rate, the full effective surface area is wetted by the scrubbing liquid. In the selection of a solvent for any particular scrubbing system, several aspects of the scrubbing liquid should be considered. These factors are: (1) the solubility of the gas should be relatively high to enhance the rate of absorption and to decrease the quantity of solvent required; (2) the solvent should have relatively low volatilities to reduce solvent loss due to evaporation; (3) the solvent should be as non-corrosive as possible to reduce the construction and maintenance costs of the equipment; (4) the solvent should be inexpensive and readily available; (5) the solvent should have relatively low viscosity to increase tower capacities and reduce flooding potentials; (6) the solvent should be non-toxic, non-flammable, chemically stable and have a low freezing point.

The diameter of a packed tower, or the gas mass flow rate per square foot of tower area, is governed either by the limiting pressure drop through the scrubber, based on blower pressure drop capacities, or on limitations on liquid build-up in the packing and/or entrainment caused by the gas. On a log-log plot of pressure drop versus mass flow rate in pounds per hour per square foot as shown in Figure 11, a plot of the data for dry-packing shows a straight line with a slope of two. As liquid flow rate is increased, the pressure drop increases. This is due to the reduction in available flow area, due to accumulation of liquid onto the packing. As the gas flow rate is increased, there is a break in the slope of the curve. This break point is called the loading point, labeled A'_1. Operation above the loading point is not normally recommended due to the increased pressure drop encountered. Further increases in the gas velocity result in a second break in the slope of the curve. This break point is called the flood point, labeled A'_2. At this condition, liquid becomes the continuous phase within the packing with the gas bubbling through.
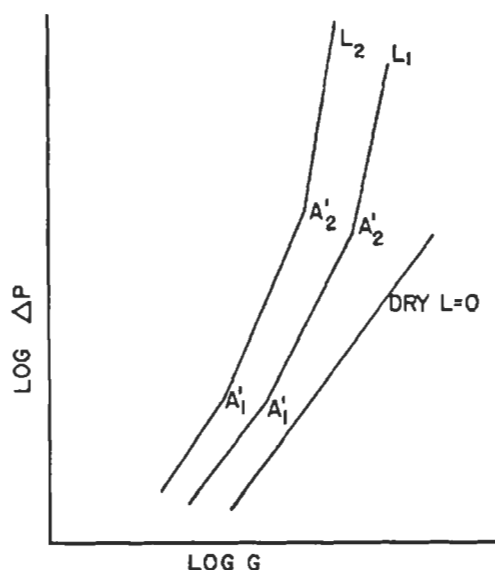
Figure 11. *Loading and flooding conditions.*

In addition to the large increases in pressure drop, high liquid entrainment into the exiting gas stream also occurs.

The packing factor (F) is an important design variable in determining the capacity of any packing design from a flooding, or pressure drop standpoint. This-factor was first described by Sherwood in 1938. The empirical correlation is:

$$F = a_p/\epsilon^3$$

where a_p is the specific area of the packing (ft^2/ft^3) and ϵ is the packing material void fraction. It has been found, however, that this approach does not take into account the effects of variations of the active area of the packing due to variations in liquid loading and packing characteristics, such as surface shielding from adjacent packing pieces, wettability, and wall and support effects. Most packing factors supplied by the various manufacturers are calculated to fit the observed flooding curves for the packing design under consideration. Further work by Eckert (see references) showed that the packing factor varied substantially for varying ratios of liquid/gas flow rates, and also for the operating pressure drop at each flow rate. Where data, such as that described by Eckert is available, they should be used for design purposes. However, published packing factors are generally adequate for most design purposes, except that there is some tendency to overdesign the tower

at high liquid/gas flow ratios, especially when Raschig Rings are used. The packing factor (F) is used in conjunction with Figure 12 to determine the estimated gas flow rates at flooding and at different pressure drops. There is no correlation establishing an estimate of the loading condition, however, for most commercially available packings, it is safe to design for gas flow rates 40 to 70% of the flooding rates calculated. Calculation of required tower diameter, based on the above flooding flow rate design criteria, can be accomplished utilizing Figure 12.

To perform the calculation, first calculate the abscissa value. Then, utilizing the flooding line curve, the ordinate value can be obtained. By solving for G, the flooding gas flow rate can be determined. By choosing an appropriate percentage of flooding gas flow rate, the tower diameter can be calculated. Alternatively, where the operating velocity is fixed by the allowable pressure drop, the appropriate pressure drop curve in Figure 12 can be used to determine the ordinate value which can then be solved for G. The ratio of operating velocity to flooding velocity can then be determined using the same procedure. The required height of the packed section of a scrubber is governed by the removal performance of the solute required of the system. In this plot, ψ is the ratio of the density of water to the density of the scrubbing liquor, μ is the solvent viscosity, ρ_L and ρ_G are the densities of the liquid and gas, respectively.
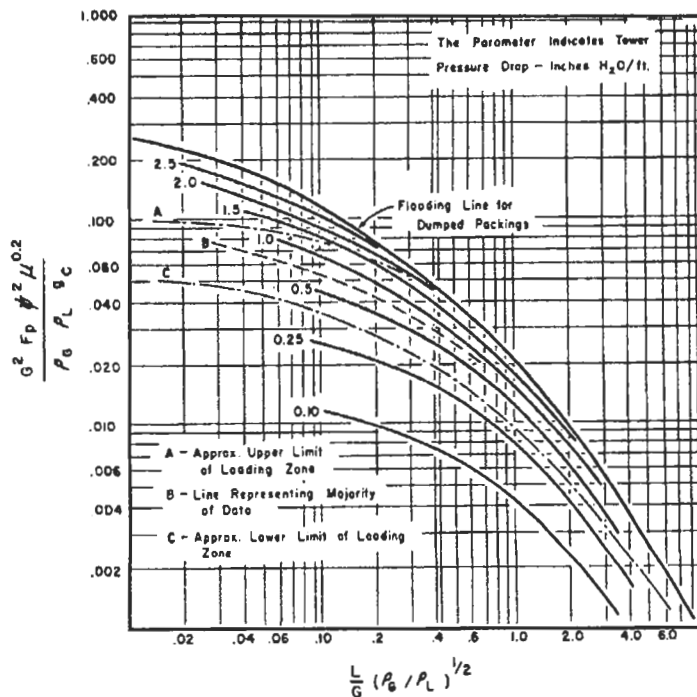
Figure 12. *Pressure drop - velocity plot.*

Going one step further, the required height of packing is governed by the rate at which absorption of the solute into the solvent takes place. This will be a function of the packing size, shape, liquid rate and liquid distribution over the surface of the packing. The height (Z) of packing required is determined by the following relation:

$$Z = H_{OG} \times N_{OG}$$

where H_{OG} is the height of a transfer unit (ft), and N_{OG} is the number of overall transfer units required. Performing a material balance over a small section of packing for the counterflow scrubber case (system definition given in Figure 13), we obtain the following over area dz:

$$d(G_m y) = K_g a(y - y^*)dz$$

From whence it follows that:

$$G_m dy/(1 - y) = \{[K_g a(1 - y)_m][y - y^*]dz / (1 - y)_m$$

subscript "m" refers to a mean value.
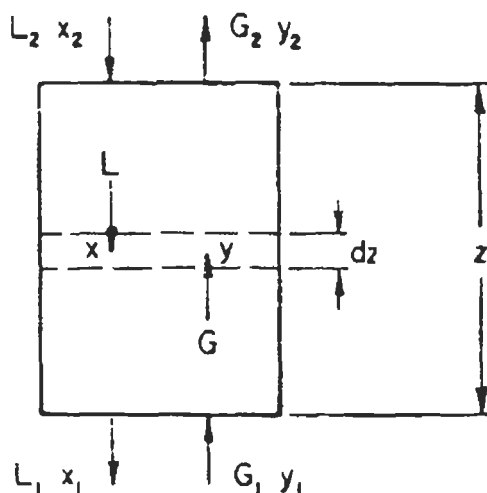
Figure 13. *Differential mass balance definition.*

Assuming that the absorption process is gas-film controlled, and that the concentration of the solute is small (i.e., $1 - y \approx 1$), then:

$$K_g = k_g$$
$$N_{OG} = N_G = \int dy/(y - y^*)$$

Introducing Henry's law ($y^* = H'x$); then the above expression reduces to the following:

$$N_{OG} = (y_1 - y_2)/(y - y^*)_{\ell m}$$

where the log-mean definition for concentration is as follows:

$$(y - y^*)_{\ell m} = [(y_1 - y^*) - (y_2 - y^*)]/[\ell n(y_1 - y^*)_1/(y_2 - y^*)_2]$$

For an absorption system in which the solute is highly soluble in the solvent, such that the equilibrium pressure of the solute over the absorbing liquid is very low, we may write:

$$y - y^* \approx y$$
$$N_{OG} = \ell n(y_1/y_2)$$

The above expression indicates that the number of overall mass transfer units, N_{OG}, is only controlled by the concentrations of the solute in the inlet and outlet gas streams.

The following tabulation of calculations shows the relationship between the number of transfer units and the percent of solute removed from the gas stream:

N_{OG}	% Absorbed $100 \times (y_1 - y_2)/y_1$	N_{OG}	% Absorbed $100 \times (y_1 - y_2)/y_1$
1	63	4	98.2
2	86.5	5	99.3
3	95.0	6	99.97

The height of a theoretical unit (H_{OG}) can be obtained from the actual process conditions whenever possible. Tower packing manufacturers have information on a wide variety of specific packing systems that enable the determination of H_{OG}. Alternatively, this information can be estimated from values of $K_g a$ using he following relationship:

$$H_{OG} = Gm/[K_g aP(1 - y)_{\ell n}]$$

Most tower packing data is based on information from standard performance test systems. These systems are usually based upon an air-feed stream with 1% CO_2 solute in the inlet stream. The scrubbing liquid entering a column is 4% NaOH in water with a carbonate conversion of 25%. Tests are normally run at a liquid temperature of approximately 75 °F. When making a selection of the specific tower packing design to be used in a scrubbing system, several packing qualities should be considered before final selection. Many of these factors interrelate with one another and various trade-offs between the subjects covered below, must be considered in the overall selection of the tower packing:

1. Cost: initial capital investment is an important consideration in the selection of any tower packing design. Costs are generally expressed in dollars per cubic foot. Generally speaking, plastic packings are less expensive than metal packing materials. However, the operating costs of the packing must also be considered. These include, power consumption, packing replacement costs, when required, solvent feed rates, and operating flexibility.

2. Corrosion resistance: it is critical to select construction materials such that neither the gas and solute nor the scrubbing liquid will corrode the packing materials. Ceramic and plastic materials, are commonly selected for this reason.

3. Temperature resistance: at high temperatures, some tower packing materials, such as plastics, may deteriorate rapidly. Further, the potential loss of cooling of upstream gases, must be considered if materials susceptible to damage by heat are considered for use. An upset might require a complete repacking of the scrubber.

4. Pressure drop: while initial installation costs are important, the continuing increase in the cost of energy makes the pressure drop consideration more and more important. The continuing operating costs involved in the selection of a high pressure drop packing, may in some cases, outweigh its initial cost advantages. Rating of existing blowers, fans, etc., when existing equipment is to be used, must be within the pressure drop limits of the packing design selected.

5. Large active exposed surface area: the specific surface area, e.g., the exposed surface area of the packing per cubic foot of packed volume, is important since larger specific area means a larger area available for contact between the liquid and the gas, resulting in more efficient scrubber operation.

6. Percent void space: high amounts of open flow area tend to reduce pressure drop, minimize the possibilities of plugging due to solids accumulation, and increase loading and flooding liquid and gas flow rates.

7. Complete utilization of the packing surface: the packing shape should be designed to prevent nesting and blockage of the surface area of one packing piece by another, thereby, reducing the effectiveness of the nested pieces for mass transfer. The packing design must also minimize liquid channeling, by forcing redistribution of the scrubbing liquid as it flows through the packing. The packing should also promote turbulent contact between the gas and liquid to allow maximum renewing of the gas and liquid at their interface. By eliminating channeling of the gas and liquid, stagnant areas, which are prone to fouling, will be eliminated.

8. Structural strength: the packing must be strong enough to withstand normal loads during shipping, installation, service, and maintenance. Also, thermal fluctuations, when present, must effect the integrity of the packing. The packing must also be strong enough to withstand the weight loads imposed by the weight of the packing resting upon it.

9. Weight: as mentioned above, weight of the packing can limit bed height. Low-weight packing, can increase the allowable packed bed depth between intermediate packing bed supports. Low side thrust loads, on the tower shell, may also be important to allow minimum tower wall thicknesses and the use of low-strength/low cost materials of construction.

10. Removal for cleaning: the tower packing should be designed for ease of removal when solids build-up may be a consideration.

11. Design flexibility: the packing should have a wide operating range with little variation in efficiency for changes in gas and liquid flow rates.

Tower packings are manufactured in a wide variety of shapes, sizes, and materials of construction. The shapes are generally broken down into two broad categories; namely, random packing and systematic packing. Random packings are installed in a non-uniform random arrangement, most often by dumping them into the retaining vessel, as described below. This type of design allows higher specific surface area, but normally results in higher pressure drop. Systematic packings are systematically stacked or laid out to provide intentionally uniform openings and surface area distribution throughout the tower packing area. These designs, in some cases, provide less surface area, but result in more uniform liquid distribution. This type

of packing design is generally more costly to install due to the substantial extra labor involved, or the higher cost of the packing itself.

Random type packings can generally be broken down into three general shape geometries: ring-type packings, saddle-type packings, and miscellaneous. The first ring type packing was the Raschig Ring, which can still be found in use in some applications, although it is relatively inefficient. Lessing rings are similar to Raschig Rings, but have a single internal cross-rib which provides strength and additional surface area for contacting. Lessing Rings are manufactured in porcelain, chemical stoneware, stainless steels, and other metals. However, with the development of newer packing designs, this packing is used on a very limited basis. Cross-partition rings are similar in geometry to the Lessing Ring, except there is a double internal cross-rib. This type of packing can serve two purposes. First, it can act as conventional tower packing. Secondly, when stacked over a series of cross bar support grids, as shown in Figure 14, in brick-lined vessels, it can act as a support for dumped packing resting upon it. These units are normally made in sizes from 3inch through 6 inch diameter from stoneware and porcelain. Larger sizes are almost always stacked. Cyclohelix spiral rings are similar to Raschig Rings, except inside the cylinder is a spiraling internal. This is normally supplied with either a single, double, or triple spiral. The purpose of the spiral is to impart a swirling, spiraling turbulence to the gas to obtain good mixing with the scrubbing liquid. This type of ring is almost always stacked rather than dumped to obtain maximum effectiveness of the spiral. Other packing designs feature openings in the walls of the ring with the resultant metal from the openings curved inward to partially simulate the cross-partition ring.
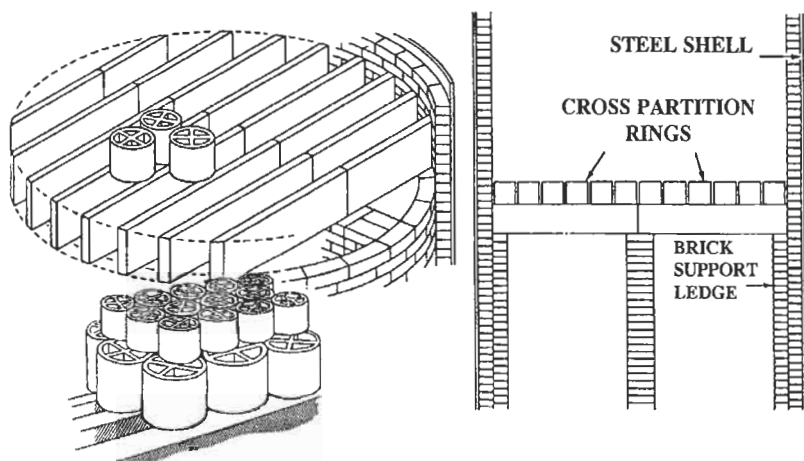
Figure 14. *Cross-partition rings used as packing supports.*

These designs, such as the Pall ring and the Flexi-ring allow much increased gas flow rates at the same pressure drop and liquid rate as the earlier Raschig Rings. This allows use of smaller diameter towers with resultant economic savings. Improved ring designs also provide higher efficiency due to a larger amount of available surface area. Also, greater gas turbulence, because of the tortuous path the gas must follow, results in better contacting.

The best known saddle-type packing is the Berl Saddle, which has the appearance of a saddle. The saddle configuration was introduced many years ago to reduce the amount of nesting and blockage of available surface area which occurred when packing vessels with Raschig Rings. Intalox saddles and Flexi-saddles are modified saddle type packing designs, improving on the basic Berl saddle configuration. Improvements in the shape further reduce the nesting or stacking which occur during installation, thereby allowing maximum surface area accessible to gas and liquid flows. These packing types are normally made in plastic, or in chemical porcelain or stoneware. Additional design variations include scalloped edges on the packing. The scallops provide many more interstitial transfer points per unit of volume than comparative packings. These transfer points continuously renew the liquid surface, greatly increasing the rate of mass transfer. The scalloped edges also serve to immobilize the packing within the bed resisting settling effects so that the initial free space and pressure drop are maintained. Helix packing are modified saddle-like packing designs normally made from polypropylene and other plastic materials. These packing designs were developed specifically for use in scrubbing type applications. Some advantages of these packing designs include: non-nesting, resulting in less channeling and increased turbulence; lower pressure drop; self cleaning design; lighter weight allows bed support savings; corrosion and heat resistant; and high mechanical strength allowing bed heights greater than 20 feet. Some miscellaneous random packing designs worth noting are the Rosette type packings, Tellerette Packing, and Spiral. Manufactured from a wide variety of plastic materials, these packing designs claim to provide many more interstitial hold-up points than can be obtained with other packing designs. These hold-up points and the use of non-wetting plastic materials create a dispersion/agglomeration cycle of the scrubbing liquor, which results in constantly fresh liquid being exposed to the gas flow.

Systematic type packings can generally be broken down into four areas, based on their internal geometries: stacked ring type, mesh type, grid type, and corrugated type. The larger diameter ring type packings can be installed by laying the rings out systematically on end, and stacking several layers one on top of the other. Ring designs used where this installation approach is considered include Raschig Rings, Lessing Rings, cross-partition rings, and cycloheilix spiral rings. This installation approach is normally used only for rings 3 inches in diameter and larger. This approach is not normally used for smaller rings because of the exponential increase in installation time required. This systematic stacking of rings requires substantial

additional labor time, resulting in much higher installation costs. Mesh and fiber-type packings have been shown to be highly efficient in absorption and distillation applications. It is in distillation, however, where these designs are most frequently used due to their extremely high performance levels. In distillation and stripping applications, the potential savings in operating costs, due to the high efficiencies, will often outweigh the packing cost. Packings are manufactured from multiple strands of wire, knitted together to form a flattened sock of mesh.

Wood grid packing designs consist of beams of wood stacked with alternate layers perpendicular to each other. This type of packing provides lower packing efficiency than the packing designs discussed to this point, however, this design is extremely economical and may be considered where low performance is required and where the wood will not be affected by the chemical environment. Drip point grids are ceramic bricks with vertical holes similar in shape to cinder blocks. When stacked in a scrubber, they provide a large surface area and high void fraction, resulting in low pressure drop. Again, performance is lower than packing designs discussed earlier. However, drip point grids are used occasionally in applications requiring particulate removal. Glitsch grids are a high open area, low pressure drop packing design, manufactured from stacked layers of perforated metal sheets which are cut to fit the vessel inside diameter. This design is considered in systems which have high solids loading as they are not susceptible to solids plugging. This packing design is frequently used in distillation columns where coking is a potential problem. It also finds use in some particulate scrubbing applications.

Corrugated packings are manufactured from relatively inexpensive metal or plastic corrugated sheets. Sheets are laid parallel to the flow with the corrugations running at a predetermined angle to the flow. Corrugations between adjacent layers of the sheet are reversed in direction, providing maximum mixing of the gas and liquid flow. The overall packing is manufactured in short length elements along the direction of the f low. These elements are stacked so that the direction of the corrugated sheets are 90° to the previous pack, creating complete mixing of the fluids over several elements. The depth of the corrugations can be varied. Higher capacity and lower pressure drop are obtained with deeper corrugations while shallower corrugations provide higher contacting efficiency. This type of packing is precut to fit inside the vessel diameter required.

Once the total bed depth of packing has been determined, the tower must be laid out so that the maximum bed depth is no more than 8 tower diameters or 20 feet maximum. Liquid distributors, to ensure uniform liquid distribution and well designed support plates, are required to realize the full potential of the packing in any application. Depending upon the size and nature of the packing being considered, the material is either stacked uniformly or dumped randomly. Where packings are installed by dumping, it is preferred that the tower be packed wet, to assure maximum randomness of the packing elements, and minimum breakage where ceramic packings are used. At least four feet of water should be kept above

the packing surface at all times for the above reasons. If possible, the water level should be up to the loading manway. When packing a large tower, the packing should be dumped at random spots, rather than developing a slope or cone packing surface, as a cone surface would tend to force the liquid flow outward to the vessel wall, creating poor liquid distribution. To get the best performance from the tower packing, it should not be shaken, dropped, walked on, or handled excessively after installation since any breakage will reduce the void area, and the efficiency. Top hold-down of the packed bed is generally recommended for plastic, ceramic, or carbon tower packing to prevent the upper portion of bed from becoming fluidized. If the packing is not held down, the resulting breakage tends to further restrict flow and encourages further surges which will ultimately destroy the packing. In this case, the hold-down rests directly on the bed and prevents bed fluidization based solely on its weight. The hold-down is not attached to the wall as this would provide no restraint once the bed had settled. For metallic packing, a bed limiter is normally substituted for the hold-down. This acts only to retain the packing inside the packing area, and to prevent it from being carried out of the vessel due to its light weight, at high pressure drops. Bed limiters are always attached to the walls of the scrubber, since breakage and crushing are not problems with these construction materials. With no moving parts, tower packing requires little maintenance during normal operations. Corrosion or solid particulate accumulation are generally the problems encountered, thereby requiring periodic replacement.

Packed tower wet scrubbers to a certain degree are considered an outdated technology, although there are still many industrial operations that utilize them throughout the world. The greatest application for scrubbers from an environmental standpoint is likely flue gas scrubbing. Many systems are packaged designs offered by vendors. An example of a packaged system is illustrated in Figure 15. In this case the scrubber cleans 97-100% of the sulphuric dioxide (SO_2, causing acid rain) and hydrochloric acid gases out of the flue gas, and also a high percentage of the dust. Since all the sulphur in the flue gas is removed, there are more options when selecting oil quality, as it becomes possible to bum waste oil and oils with a high sulphur content. The flue gas is cooled down towards 30 °C making it possible to reduce the oil consumption by up to 20% when using the scrubber. The amount of savings depends on the plant's work efficiency throughout the year and on how effectively the low temperature heat is used. The scrubber is simple, but efficient: a liquid flow meets the hot flue gas and particles, dust and water-soluble gases are washed out. The water is neutralized, cooled and reused. The water in the gas condenses when the hot gas meets the cold liquid. This process aids the removal of particles from the flue gas. In order to reduce the acidic content of the gas, lye is added to the water used in the scrubber. The salts formed can be released into the sea where they are found naturally. The scrubber is profitable for larger boiler houses for central heating, district heating, and for boilers of water or steam in industry. It is the oil consumption and the operating time, which determine the

profitability of the scrubber. The oil consumption ought to be larger than 500 tons/year in order to have a profitable scrubber plant. Standard scrubber packages that are commercially available are for oil boilers with an effect from 1.5 MW to 70 MW.

The trend in the use of gas scrubbers, particularly on small units, has shifted towards the use of non-packed columns. These are basically venturi scrubbers that were briefly described earlier in this chapter. Based on the operating principle of a liquid jet ejector, gas scrubbers are an economical means of resolving problems caused by the harmful nature of exhaust gases by scrubbing, purifying, neutralizing and eliminating them almost entirely from smoke, together with polluting odors. In some cases, the gases are a too high temperature and the scrubbing fluid can be used for cooling them. For the purification of gases having a strong smell, the scrubbing fluid has to include a chemical solution capable of interacting with the treated gas. A simplified diagram of a venturi gas scrubber is illustrated in Figure 16, in which there are three important design features, namely, the scrubbing fluid inlet configuration, the gas inlet, and the spray jet. The materials of construction used are chemically resistant, and are usually made from stainless steels, plastics, graphite, or other suitable materials.
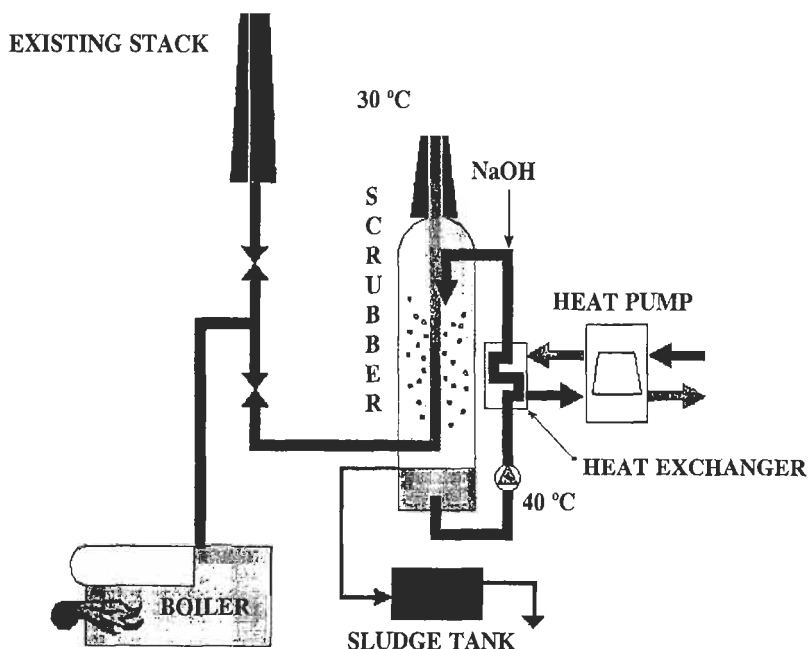
Figure 15. *Example of a packaged flue gas scrubber system.*

SCRUBBING FLUID
INLET

GAS INLET

SPRAY JET

THROAT
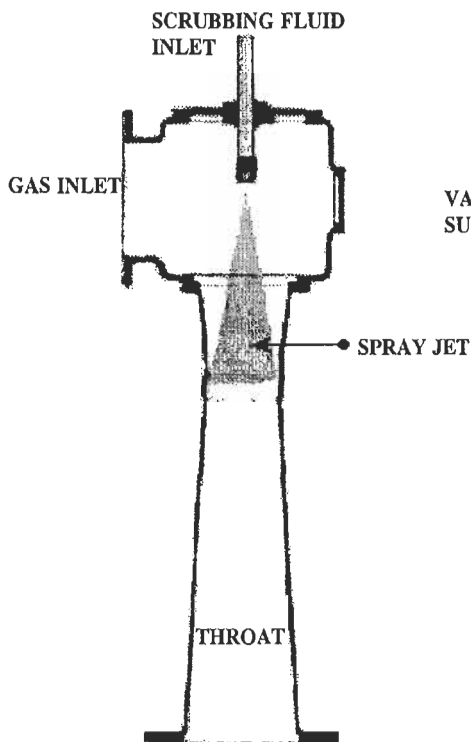
Figure 16. *Venturi scrubber.*

MOTIVE FLUID INLET

VACUUM -
SUCTION

NOZZLE

DIFFUSER

DISCHARGE
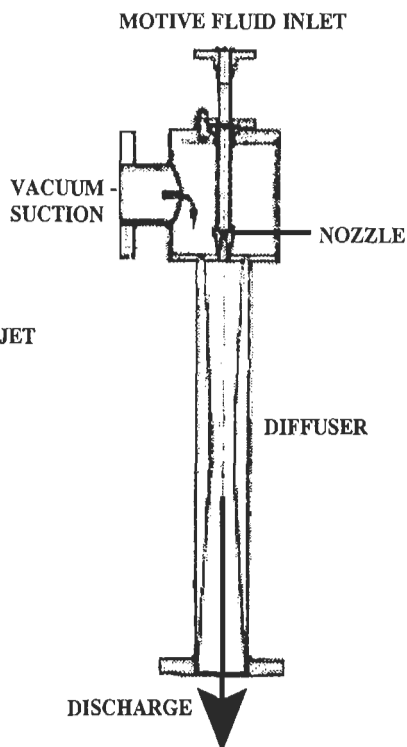
Figure 17. *Jet vacuum pump.*

Note than an ejector is the generic name of a jet appliance capable of aspirating different products: gases, liquids and solids (powders, granulates or sludge) and takes different names according to its functions: jet vacuum pump, thermocompressor, gas scrubber, eductor, etc. The operating theory is the same for every type of ejector. In the case of a jet vacuum pump, the apparatus is capable of compressing gases and fluids (optimum compression ratio: 7 to 8 = ratio between discharge pressure and suction pressure) so as to obtain constant suction pressure (more or less intense vacuum, depending on process requirements). An example of a jet vacuum pump is shown in Figure 17. Gas absorption is a unit operation that chemical engineers will certainly encounter numerous times throughout the chemical process industries. The principles outlined in this chapter provide a basic understanding of the classical design principles and practical operating guidelines. It is not uncommon for systems to be field erected for specialty applications, although again, the trend is to purchase modular or packaged units for applications such as fume scrubbing or flue gas scrubbing application. Section A of the chapter references can be consulted for additional information.

ADSORPTION

Introduction and Overview

Adsorption is the selective collection and concentration onto solid surfaces of particular types of molecules contained in a liquid or a gas. By this unit operation gases or liquids of mixed systems, even at extremely small concentrations, can be selectively captured and removed from gaseous or liquid streams using a wide variety of specific materials known as *adsorbents*. The material which is adsorbed onto the adsorbent is called the *adsorbate*. The two mechanisms involved, chemical adsorption and physical adsorption, focus specifically on carbon adsorption. When gaseous or liquid molecules reach the surface of an adsorbent and remain without any chemical reaction, the phenomenon is called *physical adsorption or physisorption*. The mechanism of physisorption may be intermolecular, electrostatic or van der Waals forces, or may depend on the physical configuration of the adsorbent such as the pore structure of the adsorbent. Physical absorbents typically have large surface areas. The properties of the material being adsorbed (molecular size, boiling point, molecular weight, and polarity) and the properties of the surface of the adsorbent (polarity, pore size, and spacing) together serve to determine the quality of adsorption. There are also the following parameters which can be used to improve physical adsorption: increase the adsorbate concentration; increase the adsorbate area; select the best absorbent for the specific gas system; remove contaminants before adsorption; reduce the adsorption temperature; increase the adsorption contact time; frequently replace or regenerate the adsorbent.

Physical adsorption units may either be regenerable types or use disposable absorbents. Regeneration of physical adsorbents is accomplished via any combination of three mechanisms, namely temperature, pressure, and concentration swings. Freshly regenerated adsorbents theoretically remove 100 percent of the contaminants and at the other extreme significant quantities of contaminants begin to escape at the breakthrough point. Physical adsorption systems may either consist of two beds (adsorption, desorption) or three beds (adsorption, desorption, cooling).

When gaseous or liquid molecules adhere to the surface of the adsorbent by means of a chemical reaction and the formation of chemical bonds, the phenomenon is called *chemical adsorption* or *chemisorption*. Heat releases of 10 to 100 kcal/g-mol are typical for chernisorption, which are much higher than the heat release for physisorption. With chemical adsorption, regeneration is often either difficult or impossible. Chemisorption usually occurs only at temperatures greater than 200 °C when the activation energy is available to make or break chemical bonds.

Activated carbon is among the most widely used adsorbent. It is usually categorized

as a physical adsorbent and also as a noppolar adsorbent. It can be produced from a wide variety of carbonaceous materials and provides an extremely high internal surface area within its intricate network of pores. A total surface area range of 450 to 1,800 m^2/gram has been estimated. Only a portion of that area is available for adsorption in pores of the proper size. For organic solvent adsorption, carbonaceous material is capable of removing at least 85 percent of the emissions. Activated carbon typically comes in three general types: granular or natural grains, pellets, and powders. The natural grains which are hard and dense are most appropriate for gaseous-phase adsorption applications while other types, liquid-phase adsorbents, are commonly used to decolorize or purify liquids and solutions. Generally, liquid-phase carbons have about the same surface areas as gas-adsorbing carbons, but have larger total pore volumes. Liquid-phase carbons are generally either powdered or granular, the former mixed and later filtered from the liquid, the latter charged into a bed. The variety of activated carbon affects what is adsorbed and how well. Activated carbon with a concentration of small pores tends to adsorb smaller molecules than large-pored carbons. The chemistry of the carbon surface and its ash constituents also affect behavior. The major application division of the carbon adsorption unit operation is between liquid-phase adsorption and gaseous-phase adsorption. Gaseous-phase carbon adsorption is primarily used for solvent vapor recovery and selective gas separations. Liquid-phase carbon adsorption is used to decolorize or purify liquids, solutions, and liquefiable materials such as waxes and in water and wastewater treatment as a polishing removal or tertiary treatment.

Carbon adsorption from the liquid phase is generally classified as a nonpolar or hydrophobic type of adsorption operation. It is generally used to remove less polar contaminants from polar bulk streams. The two basic liquid-phase equipment designs for carbon adsorption are the fixed-bed and pulsed-bed arrangements. The equipment makes use of either powdered or granular liquid-phase carbons. Fixed-bed equipment can assume the form of either single or multiple columns which can operate in series, in parallel, or both. In the typical batch configuration for a contact-batch operation, the equipment consists of an agitated tank constructed of materials suitable for the liquids being processed. The agitation allows the carbon particles to continually contact fresh portions of liquid causing mild turbulence. Where materials are sensitive to oxidation which can be caused by excessive mixing, the adsorption should be conducted under a partial vacuum or an inert atmosphere. The liquid-carbon mixture is pumped from the tank through a filter (commonly a plate and frame type). Powdered carbon is normally applied as a slurry to minimize dusting problems.

A single-column system for liquid-phase carbon adsorption is used in situations where the following conditions prevail: laboratory testing has indicated that the breakthrough curve will be steep; the extended lifetime of the carbon at normal operating conditions results in minor replacement or regeneration costs; the capital

cost of a second or third column cannot be justified due to insufficient savings in adsorbent cost; to preserve product qualities, unusual temperatures, pressure, and so on, must be maintained in the column. Unless any of these conditions prevail, a multiple-column adsorber is preferable because of the operating flexibility it provides. The choice of a multiple-column system is applicable when the nature of the process does not allow for interruption during loading, unloading, or regeneration especially when an alternate unit is not available. The choice of a multiple-column system is applicable when the nature of the process does not allow for interruption during loading, unloading, or regeneration especially when an alternate unit is not available. Multiple systems are also preferable when space constraints do not allow for a single column of adequate height or residence time.

The layouts of various fixed-bed adsorption systems and parallel fixed-bed adsorption systems are illustrated later in this section. The columns of a parallel column system are onstream at even time intervals and the column discharge is to a common manifold. The parallel design allows for smaller pumps, lower power requirements, and less stringent pressure specifications for columns and piping. Normally the adsorbent is not completely spent at the point where it is removed for regeneration. The effluent from one column of a series-column system becomes the feed for the next column. The series layout is preferred over the parallel layout if the highest possible effluent purity is desired and the breakthrough curve is gradual or if the combination of a gradual breakthrough curve and high adsorbent demand per unit of production economically needs to exhaust the carbon. The carbon in the lead column is removed during regeneration and new adsorbent is put in the column onstream at the end of the series. The former lead column is replaced by the second column. The result is that normally the operating costs for series systems are lower than for single or parallel column in the same application. Where the two layouts are combined in a series and parallel system, the best characteristics of each layout are realized.

The fixed-bed systems described can either have upward or downward liquid flows. Downflow operation has more of an inherent filtering capability. Suspended solids will be removed by the finer carbon particles at the top of the bed. The capture of significant quantities of suspended solids can lead to high-pressure drops. At this point the procedure is to backwash the adsorber which can take time and use significant wash liquid. Therefore, the downflow operation must have piping in both directions. The direction of flow is the same during adsorption and washing cycles for the upflow operations. The washing cycles are far less frequent, pressures drops are lower, and considerably less downtime and wash liquid are consumed. Although some filtration will occur, upflow operations will not produce an effluent free of turbidity or suspended solids.

Focusing discussions on carbon adsorption processes, in a pulsed-bed adsorber, the carbon moves countercurrent to the liquid. The effect is of a number of stacked, fixed-bed columns operating in series. Spent carbon is removed from the bottom of

the columns as the liquid flows upward and fresh or segmented carbon is supplemented at the top. Pulsed-bed columns are usually operated with the columns completely filled with carbon which does not allow for bed expansion during operation or cleaning. Where the pulsed-bed unit does permit bed expansion, the efficiency of the unit deteriorates due to the mixing carbon disturbing the adsorption zone. The withdrawn carbon may contain spent and partially spent carbon. Pulsed-bed adsorbers are most commonly operated on a semi-continuous basis.

During this type of operation, a set quantity of spent carbon is removed at defined intervals from the bottom of the column. Replacement of carbon is at the top. Pulsed-bed systems are the type of liquid-phase carbon adsorption which comes closest to completely exhausting the carbon with the least capital investment. The goal of either system (pulsed or fixed bed) is to maximize the use of the carbon by regenerating just the carbon that is expended. The choice of a pulsed-bed system is generally made when the feed does not contain suspended solids and the usage rate for carbon is high. Pulsed-bed systems are not effective for biologically active feeds. Conversely, fixed-bed systems are normally employed when the liquid contains significant quantities of suspended solids, is biologically active, or carbon usage is low.

Various alternative regeneration techniques for restoring spent carbon to its original adsorptive capacity make use of thermal, biological, chemical, hot gas, or solvent techniques. Multihearth or rotary furnaces can be used to volatilize and carbonize adsorbed materials. Aerobic, anaerobic, or both types of bacteria can be used on site to remove adsorbed biodegradable material. Some methods are destructive to the adsorbate and no recovery can be made. Chemical, hot gas, steam, and solvent regeneration are nondestructive methods for recovery of materials. All are carried out in place and rely on the varying adsorptive capacity of carbon for organics under changing process conditions such as pH, temperature, and nature of the liquid phase. Chemical regeneration uses a regenerant such as formaldehyde to react with the sorbed material and remove it from the carbon. Hot gas regeneration is used when carbon has adsorbed a low boiling point organic material. Steam, CO_2, or N_2 are passed through the bed causing vaporization. Solvent regeneration employs a suitable solvent to pass through the spent carbon and dissolve the adsorbed material. Solvents are then recycled and purified, usually through decantation or batch distillation. Chemical and solvent regeneration methods can be combined effectively. Steam is widely used for low-temperature regeneration.

The following are some of the typical industrial applications for liquid-phase carbon adsorption. Generally liquid-phase carbon adsorbents are used to decolorize or purify liquids, solutions, and liquefiable materials such as waxes. Specific industrial applications include the decolorization of sugar syrups; the removal of sulfurous, phenolic, and hydrocarbon contaminants from wastewater; the purification of various aqueous solutions of acids, alkalies, amines, glycols, salts, gelatin, vinegar, fruit juices, pectin, glycerol, and alcoholic spirits; dechlorination; the removal of

grease from dry-cleaning solvents and from electroplating solutions; and the removal of wastes, aniline, benzene, phenol, and camphor from water. The use of adsorption for the removal of trace contaminants, as in the commercial use for the recovery of major components of feed streams as pure products, has been a widespread application.

Gaseous-phase carbon adsorption systems can be classed in several ways. The first category is between regenerable and nonregenerable processes. The majority of industrial systems are regenerable operations that allow the user to recover the adsorbate and continue to reuse the activated carbon adsorbent. Regeneration relies on the continuity of gaseous adsorption achieved through equipment cycling to a desorption or regeneration phase of operation in which the temporarily exhausted beds of carbon are generated by removing the adsorbate. Regeneration operations are categorized in the following mechanisms: thermal swing regeneration, pressure swing regeneration, inert gas purge stripping, and displacement desorption. *Thermal swing* is widely used for regeneration in purification adsorption operations. The spent bed is heated to a level at which the adsorptive capacity is reduced so that the adsorbate leaves the activated carbon surface and is removed in a stream of purge gas. *Pressure swing* relies on the reduction of pressure at constant temperature to reduce the adsorptive capacity for an adsorbate. Pressures can drop from elevated to atmospheric or from atmospheric to vacuum conditions. *Inert purge stripping* relies on the passage of a liquid or gas, without adsorbable molecules and in which the adsorbate is soluble, through the spent carbon bed at constant temperature and pressure. *Displacement desorption* relies on the passage of a fluid containing a high concentration of an adsorbable molecule or a more strongly adsorbable molecule than the adsorbate presently on the carbon. Gaseous-phase adsorption systems are also categorized as either fixed-bed adsorbers or movable-bed adsorbers.

Fixed-Bed Carbon Adsorbers

Typical configurations of fixed-bed gaseous-phase carbon adsorption systems are illustrated in Figures 18 through 20. Enclosures for simple fixed-bed adsorbers may be vertical or horizontal, cylindrical or conical shells. Where multiple fixed-beds are needed, the usual configuration is a vertical cylindrical shell. The type of enclosure used is normally dependent on the gas volume handled and the permissible pressure drop. The gas flow can be either down or up. Downflow allows for the use of higher gas velocities, while in upflow the gas velocity must be maintained below the value which prevents carbon boiling which damages the bed. When large volumes of gas need to be handled, cylindrical horizontal vessels are selected. The beds are oriented parallel to the axis. For the continuous operation of fixed-bed adsorbers, it is desirable to have two or three units. With two adsorber

units one unit adsorbs while the other regenerates or desorbs. The required times for regeneration and cooling of the adsorbent are the factors determining the cycle time. Under most situations, two adsorbing units are sufficient if the regeneration and cooling of the second bed can be completed prior to the breakthrough of the first unit. The move to three units makes it possible for one bed to be adsorbing, one cooling, and the third regenerating. The vapor-free air from the first bed is used to cool the unit which was just regenerated. Occasionally a fourth bed is used. An example arrangement of four beds would be to have two units adsorbing in parallel, discharging exhaust to a third unit on the cooling cycle as a fourth unit is being regenerated. Figure 21 provides a schematic of a two-unit fixed-bed adsorber.
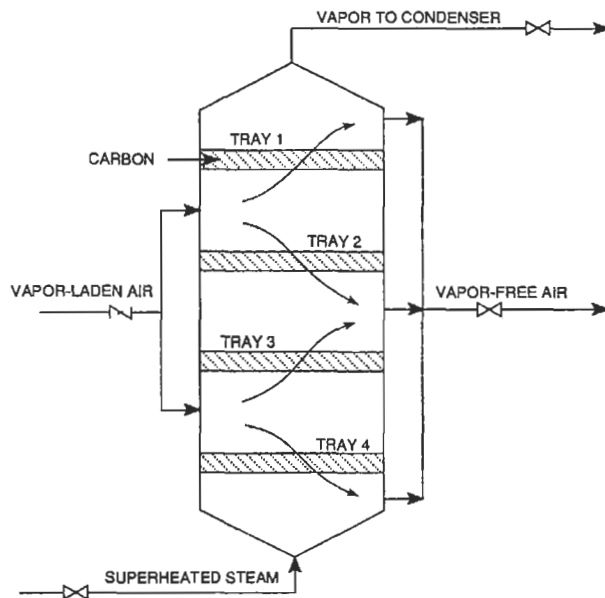
Figure 18. *Adsorber with four fixed beds.*
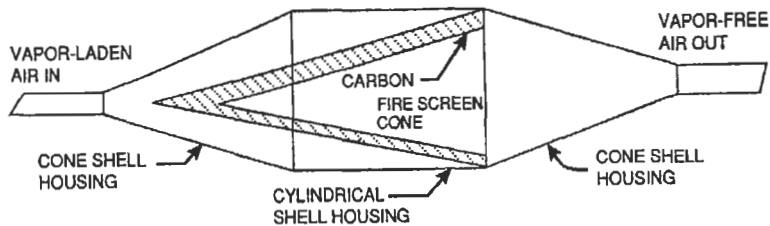
Figure 19. *Horizontal adsorber configuration.*
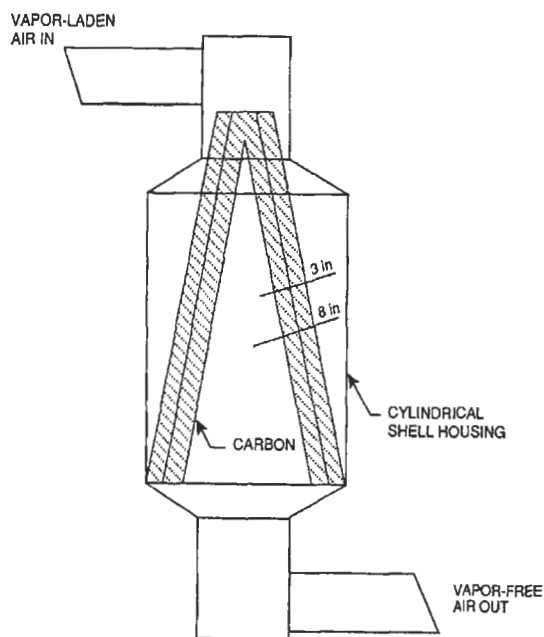
Figure 20. *Vertical adsorber with two cones.*
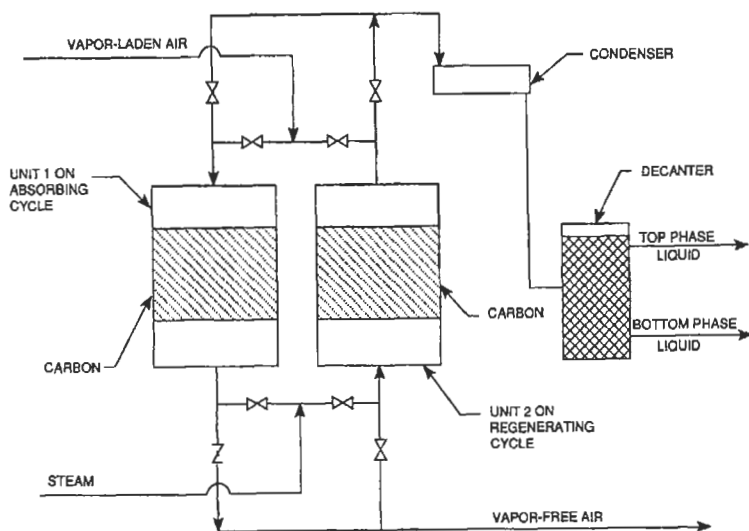
Figure 21. *Two-unit, fixed-bed adsorber.*

Conical fixed-bed adsorbers are used when a low pressure drop through the cone-shaped bed is less than one half of that through a conventional flat-bed adsorber, while the air volume is more than double that through the flat bed.

Movable-Bed Adsorbers

These are primarily used for solvent recovery and consist of a totally enclosed rotating drum housing which encloses a bed of activated carbon. A fan delivers solvent-laden air into the enclosure through ports and into the carbon section above the bed. The solvent-laden air passes through the bed to a space on the inside of the cylindrical carbon bed. The clean air discharge is through ports at the end of the drum opposite the entrance, axially to the drum and out to the atmosphere. Steam is normally used to regenerate the movable-bed adsorbers. A continuous carbon adsorber is illustrated in Figure 22.

In a typical start-up of a gas adsorption system, the operator conducts a general check of all system components: gaskets, bypass valves, adsorber and alternate unit, temperatures. Time clocks for the desired cycle times are set controlling adsorption times purge time, hot-gas drying, and cooling. Following the start of gas flow, pressure drops and temperature rises are monitored. In the case of multiple units, the operator checks to see that desorption is proceeding properly; the stripping fluid is turned on; the regeneration or disposal system for the dissolved vapors is checked; the condenser cooling-water lines are opened and the cooling-water exit temperature is monitored. During operation, temperatures and pressure drops are monitored. The prefilter is monitored to insure adequate gas flow to the processing system and is changed when a high-pressure drop is recorded. A check is made on the bed to be regenerated to make sure the proper carbon bed sequencing is set and the bed is prepared for the steam or stripping medium. When a third bed is used, a check is made to insure that this is being properly cooled. There should be a provision for routinely monitoring the adsorber emissions.
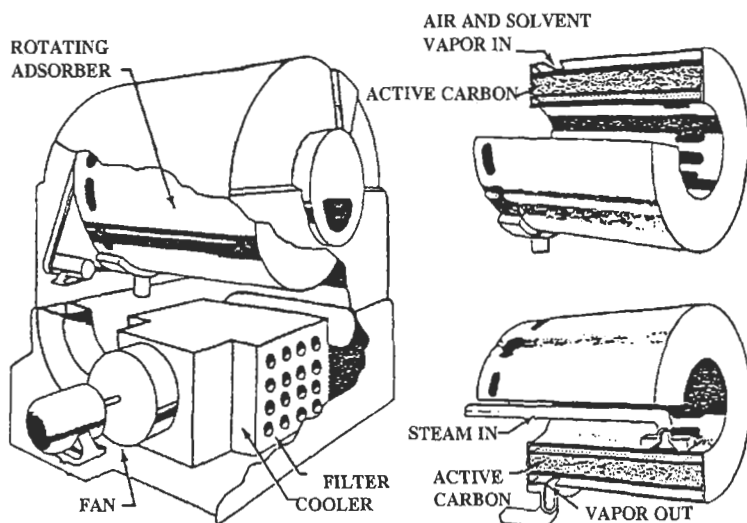
Figure 22. *Rotating fixed-bed continuous adsorber.*

Although a proper cycling procedure has been used between alternate beds, an unexpected contamination of the adsorbent would cause a premature breakthrough of the beds resulting in the release of contaminants. A routine shutdown would normally only involve the shut off of the gas flow from the process.

Gas Purification

Adsorption for gas purification comes under the category of dynamic adsorption. Where a high separation efficiency is required, the adsorption would be stopped when the breakthrough point is reached. The relationship between adsorbate concentration in the gas stream and the solid may be determined experimentally and plotted in the form of isotherms. These are usually determined under static equilibrium conditions but dynamic adsorption conditions operating in gas purification bear little relationship to these results. Isotherms indicate the affinity of the adsorbent for the adsorbate but do not relate the contact time or the amount of adsorbent required to reduce the adsorbate from one concentration to another. Factors which influence the service time of an adsorbent bed include the: grain size of the adsorbent; depth of adsorbent bed; gas velocity; temperature of gas and adsorbent; pressure of the gas stream; concentration of the adsorbates; concentration of other gas constituents which may be adsorbed at the same time; moisture content of the gas and adsorbent; concentration of substances which may polymerize or react with the adsorbent; adsorptive capacity of the adsorbent for the adsorbate over the concentration range applicable over the filter or carbon bed; efficiency of adsorbate removal required.

Complex factors enter into the process. For example, the carbon is vary rarely completely stripped of the adsorbed solvents during the regeneration process as this would require a very large quantity of steam to remove the last traces of solvent from the carbon. Also several solvents may be received simultaneously, each upsetting the equilibrium of the others. Such factors will tend to reduce the capacity of the carbon and lengthen the adsorption zone and, in order to maintain high recovery efficiency, deep-bed filters are generally used. There are no general relationships for calculating the capacity of an adsorber operating under dynamic conditions from a knowledge of the physical or chemical characteristics of the adsorbate or adsorbent. In actual practice, the adsorptive capacity, height of bed, and stripping conditions for a given adsorption application are obtained experimentally in laboratory units when the design engineer does not have applicable experience available. Dynamic adsorption tests should be conducted with gases related as closely as possible to the conditions under which the adsorption plant will operate. Whenever possible the tests should be on the gas stream to be treated containing components requiring removal.

Air pollution problems in which adsorption is considered a unit operation involve gaseous contaminants. The number of molecules present at the carbon surface is dependent on the number that reach the surface and on the residence time of these molecules on the carbon surface. If n molecules strike a unit area of a surface per unit time, and remain there for an average time, t, then a number of molecules are present per unit area of surface:

$$\sigma = nt$$

Using cm^2 as unit surface and seconds as unit time, n is the number of molecules falling on 1 cm^2/sec. The number n thus denotes the number of molecules striking each cm^2 of the surface every second, and this number can be calculated using Maxwell's and the Boyle-Gay Lussac equations. The number n is directly related to the speed of the molecules within the system. It is important to realize that the velocity of the molecules is not dependent on the pressure of the gas, but the mean free path is inversely proportional to the pressure. Thus:

$$n = 3.52 \times 10^{22} \, [p/(MwT)^{\frac{1}{2}}]$$

where: p = pressure, mm Hg

Mw = Molecular weight

T = Absolute temperature, °K

The molecular residence time, t, is difficult to determine as is the number of molecules, n.

Forces causing adsorption are the same ones that cause cohesion in solids and liquids and are responsible for the deviation of real gases from the laws of ideal gases. Basic forces causing adsorption can be divided into two groups: intermolecular or van der Waals forces, and chemical forces, which generally involve electron transfer between the solid and the gas. Depending on which of these force types plays the major role in the adsorption process, we distinguish between physical adsorption, where van der Waals or molecular interaction forces are in prevalence, and chemisorption, where heteropolar or homopolar forces cause the surface interaction. In the process of adsorption when the individuality of the adsorbed molecule (adsorbate) and the surface (adsorbent) are preserved, we have physical adsorption. If, between the adsorbate and the adsorbent, any electron transfer or sharing occurs, or if the adsorbate breaks up into atoms or radicals bound separately, then we are presented with chemisorption. The boundary layer is most important in the phase interaction. To achieve a high rate of adsorption, it

is necessary to create the maximum obtainable surface area within the solid phase. High surface area is produced by creating a large number of microcapillaries in the solid. Commercial adsorbents such as activated carbon, silica gel, alumina, and so on, are prepared in this manner. While adsorption is nearly instantaneous, the passage of molecules through capillaries (pores) may involve some time. There have been observations that in rare cases it has taken several days to reach adsorption equilibrium. The movement of molecules into the pores is a diffusion process.

Solid-gas phase interaction may occur under two conditions. One involves the random mixing of the phases; the other involves their direct relative motion. Thus, static adsorption occurs when the adsorption process takes place in relative rest, or random mechanical mixing of the phases of the solid-gas system takes place and ends in the establishment of an adsorption equilibrium among the interacting phases. Dynamic adsorption represents a sorption process accomplished under conditions of direct relative motion of one or both phases. Many applications involve dynamic conditions. Where adsorption equilibrium is not reached, it is essential to survey the equilibrium conditions because their modified effect is of major importance in dynamic non-equilibrium systems. Note that adsorption equilibrium is simply defined as the state at which the number of molecules arriving on the surface is equal to the number of molecules leaving the surface into the gas phase. The adsorbed molecules exchange energy with the structural atoms of the surface and, provided that the time of adsorption is long enough, they will be in a thermal equilibrium with the surface atoms. To leave the surface, the adsorbed molecule has to take up sufficient energy from the fluctuations of thermal energy at the surface so that the energy corresponding to the vertical component of its vibrations surpasses the holding limit.

Isotherms, as measured under existing conditions, can yield qualitative information about the adsorption process and also indicate the fraction of the surface coverage, with certain assumptions to the surface area of the adsorbent. In Figure 23, the five basic types of adsorption isotherms are presented as classified by Brunauer. Type I isotherm represents systems in which adsorption does not proceed beyond the formation of a monomolecular layer. Such an isotherm is obtained when adsorbing oxygen on carbon black at -183 °C. Type II isotherm indicates an indefinite multilayer formation after the completion of the monolayer. As an example, the adsorption of water vapor on carbon black at 30 °C results in such a curve. Type III isotherm is obtained when the amount of gas adsorbed increases without limit as its relative saturation approaches unity. The convex structure is caused by the heat of adsorption of the first layer becoming less than the heat of condensation due to molecular interaction in the monolayer. This type of isotherm is obtained when adsorbing bromine on silica gel at 20 °C. Type IV isotherm is a variation of Type II, but with a finite multilayer formation corresponding to complete filling of the capillaries. This type of isotherm is obtained by the adsorption of water vapor on

active carbon at 30 °C. The Type V isotherm is a similar variation of Type III obtained, for instance, when adsorbing water vapor on activated carbon at 100 °C. Although a large number of equations have been developed based on theoretical considerations, none of them can be generalized to describe all systems. Langmuir presented an ideal monolayer adsorption isotherm:

$$v = v_m bp/(1 + bp)$$

where: v = volumes of gas (at 0 °C and 760 mm Hg) adsorbed per unit mass of adsorbent

v_m = volumes of gas (at 0 °C, 760 mm Hg) adsorbed per unit mass of adsorbent with a layer of one molecule thick

b = empirical constant in units of reciprocal pressure

p = pressure

Brunauer further developed the Langmuir isotherm expression to include multi-layer adsorption:

$$v = v_m CX / [(1 - X)\{1 + (C - 1)X\}]$$

where v_m is taken to be a constant, C is an empirical constant, and $X = P/p_s$. This expression is referred to as the BET equation.
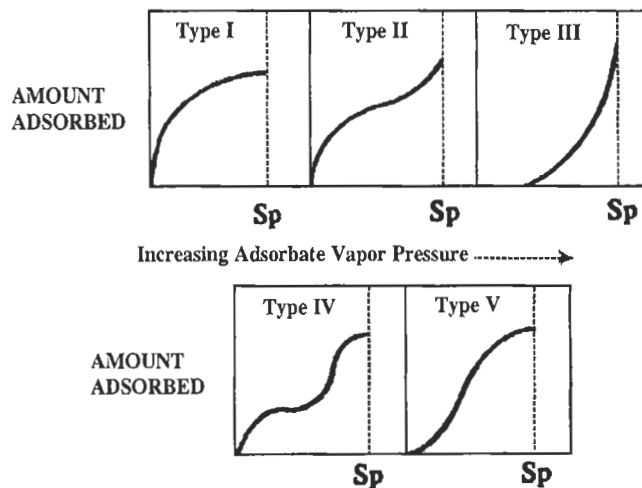
Figure 23. *Types of adsorption isotherms.*

The BET equation has two basic assumptions: (1) that the heat of adsorption is constant over the entire surface coverage of the monolayer, and (2) that the monolayer is completed before the formation of secondary layers with a heat of adsorption equaling that of the heat of liquefaction. Despite these limitations, it is very useful because it enables the numerical determination of surface area. Knowing the area occupied by a single molecule of adsorbent and the number of molecules needed to form a monolayer, it is possible to express the surface area of the adsorbent in m^2/g. Typical surface areas of commonly used adsorbents determined in this manner are: for activated alumina = 50 ~ 250 m^2/g; for silica gel = 200 ~ 600 m^2/g; for a molecular sieve = 800 ~ 1,000 m^2/g, and for activated carbon = 500 ~ 2,000 m^2/g. In most gas-solid adsorption systems, the heat of adsorption is greater than the heat of evaporation or condensation of the same substance. This means that the entropy of the molecules when adsorbed on a particular surface will be greater than the entropy of the same molecules in their liquid or solid state. In studying gas-phase adsorption, as the van der Waal forces between different molecules are approximately the geometrical mean between the values for each of the two molecules, when combined with a molecule of its own kind, it is evident that the van der Waals forces of a gas molecule on the surface of a solid will be generally greater than the van der Waals forces holding it in liquid form. There are some exceptions to the fact that the heat of adsorption is higher than the heat of liquefaction. Such is the case, for instance, when water is adsorbed on activated carbon, the polar character of the water molecule causing only weak bonds. In this case, the heat of adsorption is indeed smaller than the heat of liquefaction. Adsorption nevertheless takes place because the influence of the entropy difference is dominating. The fact that the entropy in the adsorbed state is higher than in a liquid state points to the fact that the adsorbed molecules have a greater degree of freedom than the molecules in the liquid state. Adsorption applications in gas cleaning generally involve the use of a dynamic system. The adsorbent is generally used in a fixed bed and contaminated air is passed through the adsorbent bed. Depending on the concentration, the contaminant is either recovered or discarded when the loading of the adsorbent requires regeneration. Although isotherms are indicative of the efficiency of an adsorbent for a particular adsorbate removal, they do not supply data to enable the calculation of contact time or the amount of adsorbent required to reduce the contaminant concentration below the required limits. Normal operation may be represented by Figure 24, which shows the building up of a saturated zone of adsorbers from the inlet end of the bed. As more gas is passed through adsorption proceeds, the saturated zone moves forward until the breakthrough point is reached, at which time the exit concentration begins to rise rapidly above whatever limit has been fixed as the desirable maximum adsorbate level of the fluid. If the passage of the fluid is continued on still further, the exit concentration continues to rise until it becomes substantially the same as the inlet concentration. At this point, the bed is fully saturated.
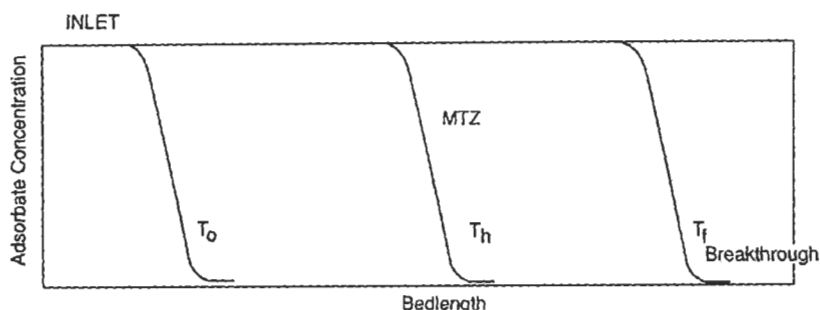
Figure 24. *Formation and movement of the MTZ through and adsorbent bed.*

While the concentration when saturated is a function of the material used and the temperature at which it is operated, the dynamic capacity is also dependent on the operating conditions, such as inlet concentration, fluid flow rate, and bed depth. The dependence of inlet concentration and fluid flow rate arise from heat effect and mass transfer rates, but the dependence on bed depth, as can be seen from the preceding description, is dependent on the relative sizes of unsaturated and saturated zones. The zone of the bed where the concentration gradient is present is often called the mass-transfer zone (MTZ). Dynamic adsorption results are expressed in terms of the dynamic capacity, or breakthrough capacity at given inlet concentrations, temperatures, and flow-rate conditions of the bed, together with the bed dimensions. It is important that the adsorber bed should be at least as long as the transfer-zone length of the key component to be adsorbed. Therefore, it is necessary to know the depth of this mass-transfer zone. Factors that play important roles in dynamic adsorption and the length and shape of the MTZ are: the type of adsorbent; the particle size of an adsorbent (may depend on maximum allowable pressure drop); the depth of the adsorbent bed and the gas velocity; the temperature of the gas stream and the adsorbent; the concentration of the contaminants to be removed; the concentration of the contaminants not to be removed, including moisture; the pressure of the system; the removal efficiency required; possible decomposition or polymerization of contaminants on the adsorbent.

Most industrial adsorbents are capable of adsorbing both organic and inorganic gases. Preferential adsorption characteristics and other physical properties make each one more or less specific for a particular application. As an example, activated alumina, silica gel, and molecular sieves will adsorb water preferentially from a gas-phase mixture of water vapor and an organic contaminant. This is a drawback in the application of these adsorbents for organic contaminant removal. Activated carbon preferentially adsorbs nonpolar organic compounds. Silica gel and activated alumina are structurally weakened by contact with liquid droplets; therefore, direct steaming cannot be used for regeneration. In some cases, none of the adsorbents has sufficient retaining adsorption capacity for a particular contaminant.
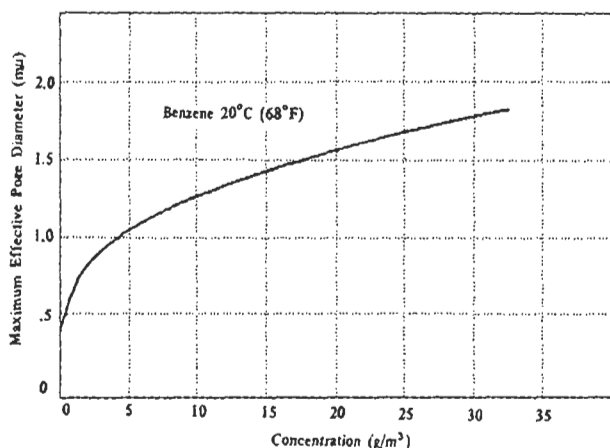
Figure 25. *Relationship between pore size and vapor concentration.*

In such applications, a large surface area adsorbent is impregnated with inorganic or, in rare cases, with a high molecular weight organic compound, which can chemically react with the particular contaminant. Iodine-impregnated carbons have been used for removal of mercury vapor, bromine-impregnated carbons for ethylene or propylene removal or recovery. The action of these impregnants is either catalytic conversion or reaction to a non-objectionable compound, or to a more easily adsorbed compound. It should be noted here that the general adsorption theory does not apply to the gross effects of the process. For example, the mercury removal by an iodine-impregnated carbon proceeds faster at a higher temperature, and a better overall efficiency can be obtained than at a low-temperature contact. An impregnated adsorbent is available for most compounds which, under particular conditions, are not easily adsorbed by non-impregnated commercial adsorbents. It is important to note that adsorption takes place at the interphase boundary; therefore, the surface area of the adsorbent is an important factor in the adsorption process. Generally, the higher the surface area of the adsorbent, the higher is its adsorption capacity for all compounds. However, the surface area has to be available in a particular pore size within the adsorbent. At low partial pressure (concentration), the surface area in the smallest pores in which the adsorbate can enter is the most efficient. At higher pressures the larger pores are becoming more important, while at very high concentrations, capillary condensation will take place within the pores, and the total micropore volume is the limiting factor. Figure 25 shows the relationship between maximum effective pore size and concentration for the adsorption of benzene vapor at 20 °C. The most valuable information concerning the adsorption capacity of a certain adsorbent is its surface area and pore-volume distribution curve in different diameter pores.

The relationship between adsorption capacity and surface area under conditions of optimum pore sizes is concentration dependent. It is very important that any evaluation of adsorption capacity be performed under actual concentration conditions. The dimensions and shape of particles affect both the pressure drop through the adsorbent bed and the rate of diffusion into the particles. Pressure drop is lowest when the adsorbent particles are spherical and uniform in size. External mass transfer increases inversely with $d^{3/2}$ (where, d is particle diameter), and the internal adsorption rate varies inversely with d^2. Pressure drop varies with the Reynolds number, and is roughly proportional to the gas velocity through the bed, and inversely proportional to the particle diameter. Assuming all other parameters being constant, adsorbent beds comprised of small particles tend to provide higher adsorption efficiencies, but at the sacrifice of higher pressure drop. This means that sharper and smaller mass-transfer zones will be achieved.

There are two effects from the adsorbent bed depth on mass transfer. First, it is important that the bed be deeper than the length of the transfer zone which is unsaturated. The second is that any multiplication of the minimum bed depth gives more than a proportionally increased capacity. Generally, it is advantageous to size the adsorbent bed to the maximum length allowed by pressure-drop considerations. The determination of the depth of the MTZ or unsaturated depth may be determined experimentally, and applying the following relationship:

$$\text{MTZ} = \quad [\text{Total Bed Depth}]/[t_2/(t_2 - t_1) - X]$$

where: t_1 = time required to reach breakpoint

t_2 = time required to saturation

X = the degree of saturation in the MTZ

The above expression may be stated in terms of capacity as follows

$$\text{MTZ} = \{1/(1 - X)\}\{D_1\}\{1 - C_1/C_s\}$$

where: D_1 = bed depth

C_1 = breakthrough capacity of bed D_1

C_s = saturation capacity

C_1 can be obtained by measuring the breakthrough capacities of two beds and using the following equation:

$$C_s = (C_2 D_2 - C_1 D_1)/(D_2 - D_1)$$

where C2 = breakthrough capacity for bed length of D_2. Direct methods for the calculation of the MTZ are also possible using transfer units; however, particularly for multicomponent systems, the calculation becomes very complicated.

Gas Velocity: The velocity of the gas stream through adsorbent beds is limited by the ad- sorbent crushing velocity and varies with different types of adsorbents. The data on crushing velocities can be obtained from manufacturers of adsorbents. As an example, the crushing velocity for a 6 × 10 mesh nutshell carbon is: V(Mw)(P) < 50,000; where V = superficial velocity, ft/min, Mw = molecular weight of the gas, and P = system pressure in atm. Crushing velocity pressure is typically less than 50,000, as determined experimentally. The length of the MTZ is directly proportional with velocity. Note that at high velocities, the unsaturated zone is elongated.

Temperature Effects: As per basic adsorption theory, adsorption decreases with increasing temperature. Because the equilibrium capacity of adsorbent is lower at higher temperatures, the dynamic or break-through capacity will also be lower, and the MTZ is proportionally changed with temperature. In some cases, refrigerated systems are used to enhance or increase adsorption. The adsorption process is exothermic. As the adsorption front moves through the bed, a temperature front also proceeds in the same direction, and some of the heat is imparted to the gas stream. When the gas leaves the adsorption front, the heat exchange will reverse and the gas will impart heat to the bed. Increase in temperature during the adiabatic operation of the adsorber bed decreases the capacity of the adsorbent. The adiabatic temperature rise in an adsorber can be calculated by assuming that there is a thermal equilibrium between the gas and the bed, and that the temperature of the outlet gas stream is essentially the same as that of the bed. Increase in temperature during the adiabatic operation of the adsorber bed can be estimated from the following expression:

$$\Delta\theta = 6.1/\{(S_g/C) \times 10^5 + 0.51 (S_A/W)\}$$

where: $\Delta\theta$ = temperature rise, °F

   W = saturation capacity of bed at $\theta + \theta_1$, °F

   C = inlet concentration, ppm

   S_g = specific heat of gas, Btu/ft^3/°F

   S_A = specific heat of adsorbent, Btu/lb/°F

Some typical values of S_A for common adsorbents under ambient conditions are: for activated carbon S_A = 0.25; alumina, S_A = 0.21; molecular sieve, S_A = 0.25.

Adsorbate Concentration: The adsorption capacity of adsorbents is directly

proportional to the concentration of the adsorbate. The concentration of the adsorbate is inversely proportional to the length of the MTZ. Thus, all else being equal, a deeper bed will be required to remove a lower concentration contaminant with equal efficiency than to remove the same contaminant at higher concentrations. It is important that for combustible gases, the concentration entering the adsorbent be kept below the lower explosive limit. The concentration and value of the contaminants also determine if recovery of the adsorbate is justified.

Presence of Contaminants/Pressure: Some portion of all gases present will be adsorbed on the adsorbent surface. Because these gases compete for the available surface area and/or pore volume, their effect will be the lowering of the adsorption capacity for the particular adsorbate, which is to be removed. Under ambient conditions, very little (10 ~ 20 ml STP/g) air is adsorbed on commercial adsorbents; however, moisture or carbon dioxide has a more significant effect. Activated carbon is less sensitive to moisture than silica gel and alumina; at high gas moisture content, its adsorption capacity can be considerably lower than adsorption from a dry air stream. It is preferred to adsorb organic contaminants from the lowest relative humidity gas stream when using unimpregnated adsorbents. The reverse is true for most impregnated adsorbents, where the moisture enhances the reaction between the gaseous contaminants and the impregnating agent. Adsorption capacity of an adsorbent increases with pressure, if the partial pressure of the contaminant increases. However, at high pressures (> 500 psig), a decrease in capacity will be observed due to retrograde condensation and a decrease in the fugacity of the more easily adsorbed compound and increased adsorption of the carrier gas. At times it is sufficient to lower the adsorbate concentration only to a small extent, while in other cases, total removal is required. Deeper adsorbent beds are required to achieve a 99.9 percent single-pass removal which is better than a partial removal efficiency of 60 percent to 80 percent. Some solvents or compounds may decompose, react, or polymerize when in contact with adsorbents. The decomposed product may be adsorbed at a lower capacity than the original substance or the decomposition product may have different corrosion or other properties. As an example, in an air stream, NO is converted to NO_2 when in contact with activated carbon. Polymerization on the adsorbent surface will significantly lower adsorption capacity and render it nonregenerable by conventional low-temperature methods such as steam. An example is the adsorption of acetylene on activated carbon at higher temperatures. Decomposition may also take place in regenerative systems during direct steam stripping of the adsorbent bed. Factors influencing adsorption which are treated individually have a combined or interrelating effect on the adsorption system. Dynamic adsorption in practice is a rather intricate process, influenced by a large number of complex factors. Some attempts have been made to develop a strictly theoretical formula for the design calculation of the adsorption system, but adsorptive capacity, MTZ, and several other factors must be experimentally determined in small-scale equipment.

Regeneration: In regeneration of a system, the main factor - economics with inplace regeneration - is or is not preferred to the replacement of the entire adsorbent charge. It is also important to establish that the recovery of the contaminant is worthwhile, or if only the generation of the adsorbent is required. If recovery is the principal objective, the best design can be based on a prior experimental test to establish the ratio of the sorbent fluid to the recoverable adsorbent at the different working capacities of the adsorbent. A typical plant, for example, will have a steam consumption in the region of 1 to 4 lb of steam per lb of recovered solvent. Under most conditions, direct steam regeneration is the most efficient. The steam entering the adsorbent bed not only introduces heat, but adsorption and capillary condensation of the water will take place, which will supply additional heat and displacement for the desorption process. The following factors should be considered when designing the stripping process:

- Length of time required for the regeneration should be as short as possible. If continuous adsorption and recovery are required, multiple sys- tems have to be installed.

- Short regeneration time requires a higher steaming rate, thus increasing the heat duty of the condenser system.

- Steaming direction should be in the opposite direction to the adsorption to prevent possible accumulation of polymerizable substances, and also to permit the shortest route for the desorbed contaminant.

- To enable a fast stripping and efficient heat transfer, it is necessary to sweep out the carrier gas from the adsorber and condenser systems as fast as possible.

- A larger fraction of the heat content of the steam is used up to heat the adsorber vessel and the adsorbent; thus, it is essential that the steam condenses quickly in the bed. The steam should contain only a slight superheat to allow condensation.

- It is advantageous to use a low-retentivity carbon to enable the adsorbate to be stripped out easily. When empirical data are not available, the following heat requirements have to be taken into consideration: (1) heat to the adsorbent and vessel, (2) heat of adsorption and specific heat of adsorbate leaving the adsorbent, (3)   latent and specific heat of water vapor accompanying the adsorbate, (4) heat in condensed, indirect steam, (5) radiation and convection heat losses.

Since the adsorbent bed must be heated in a relatively short time to reactivation temperature, it is necessary that the reactivation steam rate calculation is increased by some factor that will correct for the nonsteady-state heat transfer. During the steaming period, condensation and adsorption will take place in the adsorbent bed, increasing the moisture content of the adsorbent. A certain portion of the adsorbate

will remain on the carbon. This fraction is generally referred to as heel. To achieve the minimum efficiency drop for the successive adsorbent cycles, the adsorbent bed should be dried and cooled before being returned to the adsorption cycle. The desired state of dryness will depend on the physical properties of the adsorbate and on the concentration of the adsorbate in the carrier stream. When using high adsorbate concentrations, it may be desirable to leave some moisture in the adsorbent so that the heat of adsorption may be used in evaporating the moisture from the adsorbent, thus preventing any undue temperature rise of the adsorbent bed. It is also necessary to establish the materials of construction on the basis that several compounds, especially chlorinated hydrocarbons, will undergo a partial decomposition during regeneration, forming hydrochloric acid. Safety factors have to be considered in designing a regeneration system, assuring that the adsorber is not being used at temperatures higher than the self-ignition point of the contaminant. Carbon does not lower the ignition temperature of solvents and, as an example, solvent adsorbed on carbon ignites at the same temperature as the solvent vapor alone.

Activated Carbon versus Molecular Sieves: The process of activation using activated carbon can be described as slow dehydration and carbonization, usually effected by heating the raw material in the absence of air. Experimental work has shown that activation of carbon can only occur on chars that have been made at temperatures below 600 to 700 °C. The activation process selectively enlarges the pores of the carbon to provide high adsorptive capacity. The surface of activated carbon presents a largely homogeneous distribution of electrical charge. For this reason, activated carbon does not show any preferential adsorption of polar molecules such as water, but rather will desorb polar materials in favor of nonpolar or materials of higher molecular weight. Thus, a nonpolar solvent would be preferentially adsorbed in a water- solvent mixture. Because there are many types and grades of activated carbon available commercially, it has become necessary to establish specifications to obtain the proper carbon. The activity and retentivity of the carbon are generally based on their ability to adsorb a standard CCl_4 solvent. A typical set of carbon specifications is shown in Table 2. Zeolites are crystalline aluminosilicate minerals. A zeolite is an alummosilicate with a framework structure endorsing cavities occupied by large ions and water molecules, both of which have considerable freedom of movement, permitting ion exchange and reversible dehydration.

Activation of zeolites is a dehydration process accomplished by the application of heat in a high vacuum. Some zeolite crystals show behavior opposite to that of activated carbon in that they selectively adsorb water in the presence of nonpolar solvents. Zeolites can be made to have specific pore sizes that will increase their selective nature due to the size and orientation of the molecules to be adsorbed. Molecules above a specific size could not enter the pores and therefore would not be adsorbed.

Table 2. *Examples of Specifications for Activated Carbon.*

Property	Specification
Activity for CCl_4[a]	At least 50 %
Retentivity for CCl_4[b]	At least 30 %
Apparent Density	At least 0.4 g/ml
Hardness (Ball Abrasion)[c]	At least 80 %
Mesh Distribution	6 - 14 range
	(Tyler sieve series)

a - Maximum saturation of carbon at 20 °C and 760 torr in air equilibrated with CCl_4 at 0 °C;
b - Maximum weight of adsorbed CCl_4 retained by carbon upon exposure to pure air at 20 °C
and 760 torr; c- Percent of 6 - 8 mesh carbon which remains on a 14-mesh screen after
shaking with 30 steel balls of 0.25 - 0.27 in. diameter per 50 g carbon for 30 min. on a
vibrating or tapping machine.

Engineering Considerations: To effect the good engineering design of an activated carbon adsorption system, it is first necessary to obtain information on the following: the actual cubic feet per minute (ACFM) of air to be processed by the adsorber, the temperature of gas stream, the material(s) to be absorbed, the concentration of the material to be adsorbed, and if the intended application is air pollution control such as odor control - then the odor threshold of the material to be adsorbed. In addition, data is needed on the presence of other constituents in the gas stream, and whether or not solvent recovery is economical.

Once this information has been obtained, the cyclic time of the system can be established. This is primarily an economic consideration, and should be reviewed and reevaluated after the initial sizing of the system. Should the initial capital cost of the adsorber be too high, the cyclic time may be reduced to enable use of a smaller system. In general, the larger the system, the greater the overall efficiency, and the less energy that will be spent per pound of material adsorbed.

The normal starting point would be to select a half working shift cyclic time so that the unit changeover could be made during a working break. The weight of adsorbent required is then determined using the following equation:

$$W = t \, e \, Q_r Mw \, C_v / 6.43 (1 \, 0)^6 S$$

where: t = duration of adsorbent service before saturation (hr)

e = sorption efficiency (fractional)

Q_r = air flow rate through the sorbent bed (ACM)

Mw = average molecular weight of the sorbed vapor

C_v = entering vapor concentration (ppm by volume)

S = proportionate saturation of sorbent (fractional)

Table 3 provides typical maximum values for retentivities under standard conditions. The sorption efficiency *e* is a variable determined by the characteristics of the particular system, including concentration and temperature. For the purposes of engineering design calculations, it is normally assumed to be unity.

The design engineer must also control the inlet temperature to be less than 100 °F at the inlet to the unit. The next step is to calculate the volume of carbon required based on the bulk density of the carbon:

$$V_{bed} = W/D_c$$

Where D_c is bulk density. An equation for the overall pressure drop of the system based on the area of the bed, A_b, is:

$$A_b = Q_r/V_S$$

where V_S is the superficial linear velocity of gas through the bed, expressed in terms of the height of the bed, H_b, as: $H_b = V_{bed}/A_b$. From whence, the total pressure drop across the bed is: $\Delta P_T = V_{bed}V_s/Q_r$. The system pressure drop is based on an economic balance between the size of the system needed for a specific velocity, and the power requirements needed for the ΔP_T.

Once ΔP_T is established, the height and area of the bed can be calculated from earlier expressions, and then one can select a cylindrical tank or pressure vessel. Should the objective of the absorption process be solvent recovery, then the amount of solvent retained in the bed must be calculated in order to determine how much steam is needed for stripping purposes. The amount (or weight) of solvent is simply equal to S × W.

Solvent recovery systems would also necessitate the specification of condenser duties, distillation tower sizes, holding tanks, piping, and valves. It is important to note that the engineering design of an adsorption system should be based on pilot data for the particular system. Information can usually be obtained directly from the adsorbent manufacturer. The overall size of the unit is determined primarily by economic considerations, balancing the operating costs against the capital costs.

Adsorption, as can readily be seen, is not an exact science, but rather an art that draws on the experience of the design engineer. Various adsorber recovery system operations are illustrated in Figures 26 and 27.

These simplified schematics provide an overall view of the types of operations, equipment and configurations typically employed in adsorption operations. Additional discussions on these systems can be found among the references cited in Section (B) of the suggested readings at the end of this chapter.

Table 3. *Retentivity of Gases by Activated Carbon.*[†]

Gas	Retentivity (%)	Gas	Retentivity (%)
Formaldehyde	nil	Methyl ether	10
Formic acid	7	Methyl ethyl ketone	25
Heptane	23	Methyl isobutyl ketone	30
Hexane	16	Methyl mercaptan	20
Hydrogen Bromide	12	Methylene chloride	25
Hydrogen chloride	12	Naphthalene	30
Hydrogen fluoride	10	Nicotine	25
Hydrogen iodine	15	Nitric acid	20
Hydrogen sulfide	3	Nitro benzene	20
Iodine	40	Nitrogen dioxide	10
Iodoform	30	Nonane	25
Isopropyl acetate	23	Octane	25
Isopropyl alcohol	26	Palmitic acid	35
Isopropyl chloride	20	Pentane	12
Isopropyl ether	18	Phenol	30
Methanol	20	Propane	5
Methyl acetate	16	Propionic acid	30
Methyl alcohol	16	Propylene	5
Methyl chloride	5	Pyridine	25
Turpentine	32	Sulfuric acid	32

† *Data reported are percent retained in a dry air stream at 20 °C, 760 torr pressure.*

Solvent Recovery: We now focus attention on the operation of recovery. Volatile solvents vaporized during a manufacturing process may be recovered and used again. From the mixture of air and vapor, which is generally the form in which the solvent must be sought, the latter may be condensed to a liquid and trapped by the application of cold and moderate pressure; the vapor-laden air may be passed

through a liquid adsorbent such as water; or finally, the mixture may be passed through a sufficiently thick bed of a solid adsorbent such as activated carbon and later driven off by steam. There are certain conditions which each of these processes meets better than the other two. Condensation by cooling may be properly selected when the concentration of the vapor is very high; adsorption in oil in a long series of plate towers has been the general practice for separating natural gasoline from hydrocarbon gases; in the general chemical and allied industries, adsorption on activated carbon is favored. An installation for continuous operation consists of a blower, two adsorbers, a condenser, a decanting vessel to separate solvents which are im- miscible with water, and more or less elaborate rectification or distillation equipment for solvents which are miscible with water.
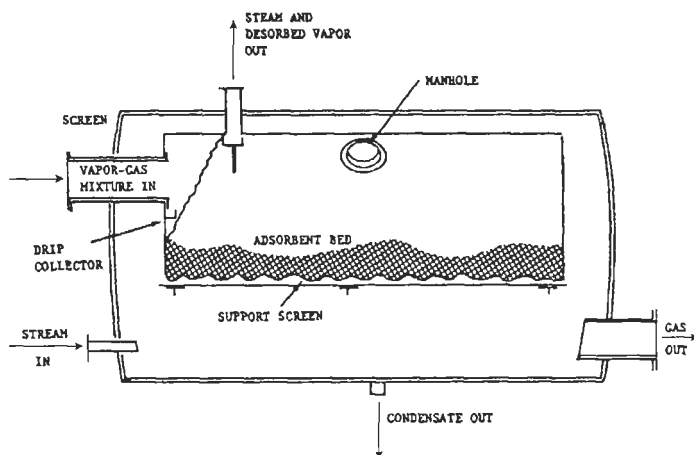
Figure 26. *Activated carbon bed adsorber*.

A - SOLVENT LADEN AIR INLET   E - COOLING WATER RETURN
B - STRIPPED AIR OUTLET   F - RECOVERED SOLVENT OUTLET
C - STEAM INLET   G - EFFLUENT TO DRAIN
D - COOLING WATER SUPPLY   H - CONDENSATE TO DRAIN
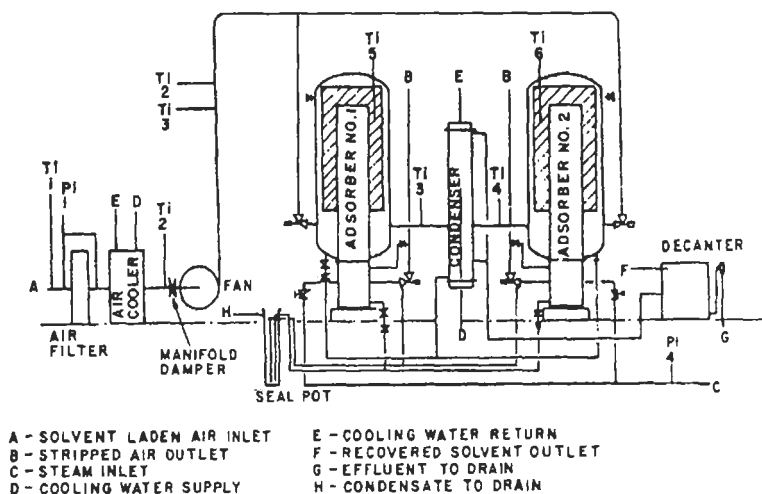
Figure 27. *Preliminary flow sheet of solvent recovery plant*.
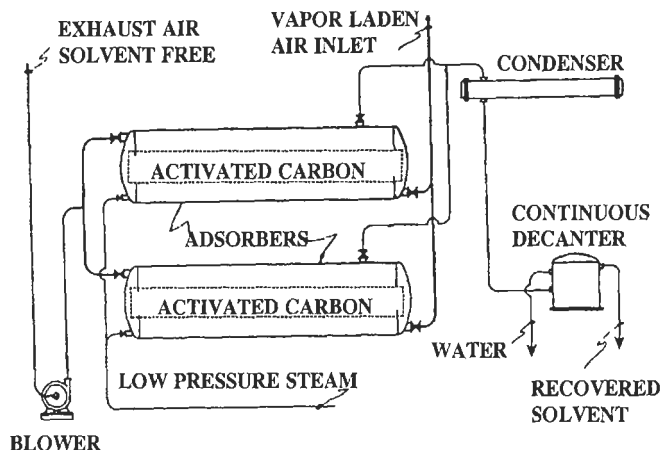
Figure 28. *Activated carbon solvent recovery plant.*

The adsorbers are usually built of steel, and may be lagged or left unlagged; the horizontal type is shown in Figure 28. The vapor-laden air is fed by the blower into one adsorber which contains a bed of 6- to 8-mesh activated carbon granules 12 to 30 inches thick. The air velocity through the bed is 40 to 90 feet per minute. The carbon particles retain the vapor; only the denuded air reaches the exit, and then the exhaust line. The adsorption is allowed to continue until the carbon is saturated, when the vapor-laden air is diverted to the second adsorber, while the first adsorber receives low-pressure steam fed in below the carbon bed. The vapor is reformed and carried out by the steam. The two are condensed and if the solvent is not miscible with water, it may be decanted continuously while the water is run off similarly. After a period which may be approximately 30 or 60 minutes, all the vapor has been removed, the adsorbing power of the charcoal has been restored, and the adsorber is ready to function again, while adsorber No. 2 is steamed in turn.

The life of the carbon will depend on the type of carbon employed, on the solvents and impurities passed to it, and on the operating conditions. The system may be modified to provide, in addition, a cooling and partial drying of the carbon bed after steaming, so that it is not placed in service again until cool and partially dried. The opening and closing of the valves which divert the vapor-laden air from one adsorber to the other and the corresponding connection and disconnection to the exhaust line may be done manually or automatically. When the rate at which the air is fed in is constant, and likewise its vapor content, the system may be operated automatically on the basis of time. But even when the composition of the vapor-air mixture varies, automatic operation is available by using a vapor detector in the exhausted air. When the activated carbon in adsorber No. 1 is saturated, some vapor escapes, is detected, and the operation of an electrical device closes the inlet valve and opens or closes all other valves in the predetermined direction and order,

so as to end the air mixture to adsorber No. 2. The system is then fully automatic. Solvents which have been successfully recovered by the activated carbon adsorption method include methanol, ethanol, butanol, chlorinated hydrocarbons including perchlorethylene, which boils at 121 °C (250 °F), ethyl ether, isopropyl ether, the acetates up to amyl acetate, benzene, toluene, xylene, mineral spirits, naphtha, gasoline, acetone, methyl ethyl ketone, hexane, carbon disulfide, and others.

The volatile solvents recoverable by the activated carbon system or any other system are nearly all organic, and many of them form flammable or explosive mixtures with air. Such mixtures may lie between upper and lower explosive limits. The activated carbon system can avoid the explosive range by staying well below the lowest percentage of vapor which is still explosive; it functions well at very low concentrations. The system also recovers solvents efficiently even in the presence of water; the recovery efficiency is high (98 percent and 99 percent are not unusual); it may be fully automatic. The annual maintenance charge rarely exceeds 5 percent of the cost of equipment. The recovery expense may be as low as 0.2 cent per pound in some installations; it rarely exceeds 1 cent per pound.

The recovery of solvents by direct condensation is usually practiced where pure solvent vapors, or solvent vapors at high concentrations are encountered. The method of cooling may be indirect by the use of a heat exchanger or by bringing the vapors in direct contact with the cooling medium (e.g., in contact with water in a jet condenser). Examples of applications using condensation as a means of solvent recovery are dry-cleaning machines, coating machines, and solvent drying and printing operations.

Treatment of Liquids

Carbon treatment has been used in numerous positions in the sequence of plant operations. Sometimes, it is used early in a process to remove gross quantities of a contaminant or it has been used as a final step for improving product quality by removing trace components and as a polishing operation, or in wastewater treatment as a tertiary or advanced treatment process. Both powered and granulated activated carbon have been used successfully and there are advantages and disadvantages to using each type. However, there has been a trend to convert existing powdered carbon operations to granular systems and to use granular carbon in new systems. Reasons for this are: decreasing price differential between the two carbons; problems with disposal of powdered carbon and filter cake; lack of a competitive thermal reactivating system for powdered carbon; greater labor requirement for the powdered carbon system; higher product losses per weight of carbon used; inefficient use of the carbon. On a theoretical basis, it is not possible to predict how effective carbon will be in treating a given liquid, or the conditions under which the

carbon will do the most effective job. Therefore, it is usually necessary to conduct laboratory tests to determine: (1) to what extent removal of the component(s) or adsorbate is possible; (2) carbon dosage, or g carbon/g of liquid; (3) the amount of carbon required to be onstrearn to efficiently remove the adsorbate; (4) the effect of linear flow rate and superficial contact time on performance of the carbon; (5) the type of system to install, that is, fixed beds or pulse beds - singly, in series, or in parallel; (6) the effect of temperature and/or pH on the adsorption capacity. A complete laboratory investigation would generally consist of two parts: preliminary isotherm tests would be performed to demonstrate the feasibility of granular carbon treatment and laboratory column tests would be conducted to obtain data to be used in designing the full-scale plant. Unlike column tests in water-related applications, which may take several months, most column runs for chemical applications can be completed in less than a month.

An adsorption isotherm is a simple method of determining the feasibility of using granular activated carbon for a particular application. A liquid-phase isotherm shows the distribution of adsorbate (that which is adsorbed) between the adsorbed phase and the solution phase at equilibrium. It is a plot of the amount of adsorbate adsorbed per unit weight of carbon versus the concentration of the adsorbate remaining in solution. Straight-line plots can generally be obtained by making use of the empirical Freundlich equation, which relates the amount of adsorbate in the solution phase to that in the adsorbed phase by the expression:

$$\Phi/m = kC^{1/n}$$

where: Φ = amount of adsorbate adsorbed

m = weight of carbon

Φ/m = concentration in the adsorbed phase, that is, the amount of adsorbate adsorbed per unit weight of carbon

C = equilibrium concentration of adsorbate in solution after adsorption

k, n = empirical constants

Taking the logarithm of both sides we obtain:

$$\log(\Phi/m) = \log k + 1/n \log C$$

This equation is a straight line whose slope is k at C = 1. Therefore, if Φ/m is plotted against C on log-log paper, a straight line will normally be obtained. However, there are occasions, as explained later, when this is not true. The straight and the curved isotherm lines provide valuable information for predicting adsorption operations.

In liquid-phase applications, transfer of the adsorbate from the bulk solution to the carbon particle must proceed through two stages: first, via transfer of the adsorbate from the bulk liquid to the surface of the carbon particle, and second, by migration

of the adsorbate from the surface of the carbon to the adsorption site within the particle. Kinetic experiments demonstrate that the transfer of adsorbate is appreciably more rapid than the migration of the absorbate; so the latter is normally the predominant rate determining step. Note that the rate of adsorption varies with the diameter of the carbon particles used in tests.

To increase rates of adsorption and decrease the time necessary to complete the isotherm, it is recommended that the granular carbon be pulverized so that 95 wt % will pass through a 325-mesh screen. Such pulverization does not significantly increase the surface area. The increase in the surface area, in most cases, is less than 1 percent, as the vast majority of surface area is contributed by the pore walls rather than by the external surface of the carbon particles.

Adsorption efficiency is usually a function of system temperature. For laboratory evaluation of granular activated carbon of liquid-phase applications, it is recommended that the temperature of the existing process stream be determined first. If the desired degree of adsorption occurs at this temperature, then the plant operating parameters will not have to be altered. If, on the other hand, the desired degree of adsorption does not take place at the existing process temperature, then a higher or lower temperature should be evaluated. The selection of the temperature will depend on such parameters as viscosity, the thermal stability characteristics of the test liquid, and the feasibility of changing the process temperature in the plant.

Adsorption capacity can also be a function of the pH of the liquid. Normally, when performing laboratory evaluations, the pH of the process stream is used. If the desired degree of adsorption does not take place at the process pH, then various pH levels should be investigated. Care must be taken when adjusting the pH of a process stream to make sure that the change in pH does not degrade or decompose a particular product or material. When treating liquids with a low pH, it is advisable to use an acid washed carbon, since a portion of the ash constituents of non-acid washed activated carbon will be solubilized under acidic conditions. Contact time is critical to the adsorption process. It should be sufficiently long to allow an approach to adsorption equilibrium. A preliminary experiment can be performed to determine the contact time required to attain equilibrium. Normally, 0.5 gram portions of pulverized activated carbon should be added to several 100-g portions of the test liquid (when the density of the liquid is near that of water, 100 ml of the liquid, carefully measured, may be used) and each portion agitated for various time periods at the process temperature and pH. For example, portion 1 is agitated for 30 minutes portion 2 for one hour, portion 3 for two hours, and so forth. At the end of each contact time, the carbon is removed by a suitable means such as filtration or centrifugation. If the quantity of adsorbate remaining in solution is plotted as a function of time, a curve similar to Figure 29 should be obtained. It is apparent from Figure 29 that the contact time required to reach equilibrium for this particular liquid is about three hours. Isotherm experiments for estimating the required carbon dosage then must be performed with a minimum of three hours contact time.
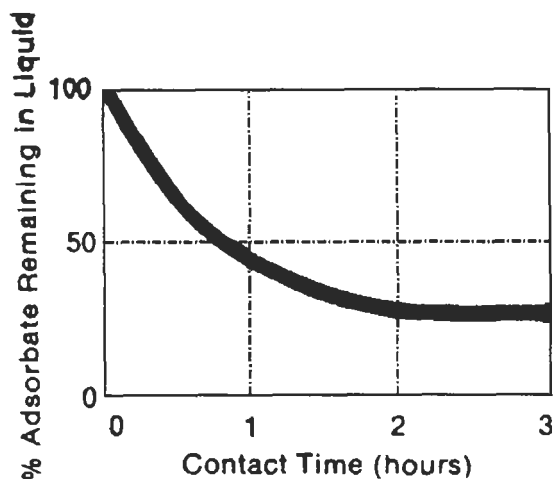
Figure 29. *Typical contact time curve.*

To obtain a meaningful isotherm, as wide a range of carbon dosages as practical should be used. Recommended dosages are 0.05, 0.1, 0.2, 0.5, 1.0, 2.5, 5.0, and 10.0 g of carbon per 100 g of the test liquid. Prior to analysis of the treated liquid, the carbon must be removed from the liquid. Carbon can be removed by filtration. The filtration rate may be increased by using heat or pressure. If the liquid is so viscous that filtration is difficult, centrifugation can sometimes be used as a suitable separation.

After the carbon has been removed from the treated liquid, the amount of adsorbate remaining in solution can be determined. Any analytical method or instrument that can determine the quantity of the particular adsorbate remaining in the test liquid may be used. Techniques include titration, spectrophotometry, gas/liquid chromatography, and total organic carbon analysis. The isotherm will determine whether the desired degree of adsorbate removal is possible with the particular activated carbon tested. If a vertical line is drawn from the point on the horizontal axis corresponding to the influent concentration (C_o) line, and the best line through the data is extrapolated to intersect this (C_o) line, the (Φ/m) value at the point of intersection can be read from the vertical scale. This (Φ/m) value represents the amount of adsorbate adsorbed per unit weight of carbon when that carbon is in equilibrium with the influent concentration. This is the ultimate capacity of the carbon at these conditions. The ultimate capacity may or may not be realized in column operation, since the adsorption rate is limited. Thus, the superficial contact time in a column operation is important. From the value of (Φ/m), the quantity of liquid treated can be calculated using the following formula:

$$W_{C_o} = (\Phi/m) \times (W)/C_o$$

where: W_{C_o} = theoretical weight of liquid treated per unit weight of carbon

Φ/m = capacity per unit weight of carbon at the influent concentration

W = weight of liquid used in the isotherm test

C_o = influent concentration

If the liquid was measured by volume, the same calculation should be used by substituting V_{Co} and V in the proper places.

$$V_{co} = (\Phi/m) (V)/C_o$$

where: V_{co} = theoretical volume of liquid treated per unit weight of carbon

V = volume of liquid used in the isotherm test

The performance of two or more carbons in the same application can be compared by examining their isotherms. Usually, granular carbon with the higher Φ/m value would be preferred for an application.

Ideally, straight-line isotherm plots are obtained. However there may occasionally be departures from linearity. A curve as shown in Figure 30 (A) may be obtained if a non-adsorbable impurity is present in the liquid being treated. For such situations subtracting C_1 from C_o and re-plotting the isotherm will usually yield a straight line.
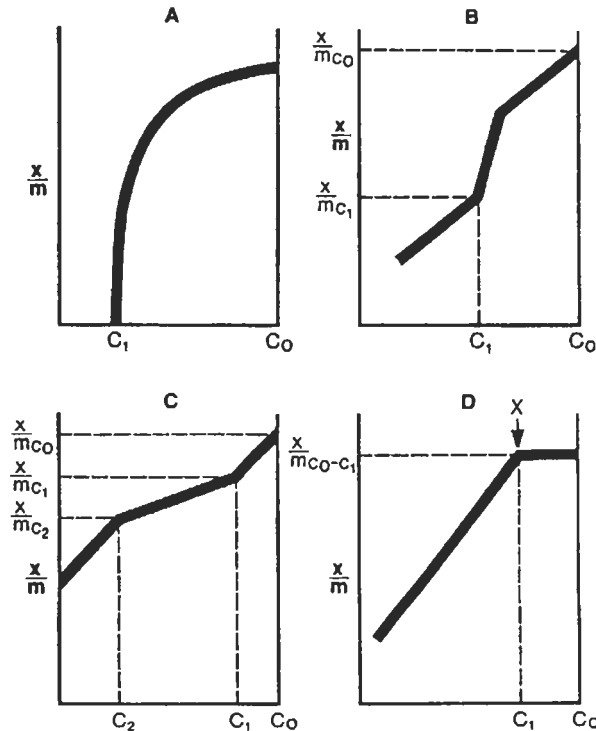
Figure 30. *Examples of non-linear isotherms.*

A sudden change in slope (as in Figure 30) indicates two components (adsorbates) are present which are adsorbed at different rates with markedly different capacities. Figure 30 (C), similar to that in Figure 30 (B), illustrates a situation in which three compounds (adsorbates) are present, and all are adsorbed in different degrees. When many adsorbate species are present, but all are adsorbed equally, straight-line isotherms still will be obtained. Figure 30 (D) indicates that the adsorbate has reached its maximum surface coverage at capacity X. Therefore, any increase in concentration of adsorbate does not result in increased capacity. From an isotherm test, it is possible to determine whether a required adsorbate removal can be accomplished and obtain the ultimate capacity of the granular carbon for that application. From the capacity figure, an estimate of the minimum granular carbon usage rate necessary to meet the treatment objective can usually be obtained. Isotherm tests also afford a convenient method for comparing different carbons and for investigating the effects of pH and temperature. The lowest possible carbon usage rate is predicted by a properly run isotherm test. If a lower usage is observed in column tests or in the plant, then one or more of the following is true: (1) the isotherm test was not at equilibrium; (2) the interpretation of the isotherm was done incorrectly; (3) there is an error in the analytical techniques; (4) the carbon fines may not have been completely removed from the solution; and/or liquids tested in the isotherm test and in the column tests were not identical. If isotherm studies indicate that the liquid can be treated to the desired purity level at a reasonable dosage, then the next step is to evaluate the liquid in a dynamic test.

Adsorption on granular activated carbon is a diffusion process consisting of the following steps: (1) bulk diffusion of the adsorbate from the liquid to the film around the carbon particle; (2) diffusion through the film; and (3) internal pore diffusion to the adsorption sites. In multicomponent systems there is the added step of competition for site results in displacement of previously adsorbed, but less strongly held, components. Ideally, it would be desirable to be able to mathematically model the carbon performance from the equilibrium data. In studies used to develop models, one or more of the following elements is usually present in the study or assumed in the model: uniform carbon particle size, ideal isotherm, dilute solutions, , single component, adsorbate molecule was too large to enter any pore, so there was no pore diffusion, thus film diffusion was controlling, the flow rate was high so pore diffusion was controlling, comparisons of predicted versus actual data were made on the early part of the breakthrough curve before the carbon was saturated, and/or systems with relatively short mass-transfer zones (MTZ) were studied. There is no model presently available that can be used universally. Until such time as one is developed, it will be necessary to test each liquid in a dynamic system and use empirical methods to design the plant-scale units. Generally, the tests can be controlled better in the laboratory as opposed to in-plant testing. However, tests should be conducted in the plant if any of the following conditions exist: the liquid to be treated would deteriorate or change in characteristics during

shipment and/or storage; carbon dosage is so low that large quantities of liquid would be required to conduct the test; properties of the liquid stream being treated vary widely. This is usually not nearly as important a factor as it is in wastewater applications. Generally, experience may serve as a guide to selecting the size of the system. If there is no previous history for the liquid, it is best to overdesign the capacity. In the laboratory, the columns are usually 2.5-5.0 cm in diameter by 1-1.5 meters deep. In the plants, the size is usually 5-30 cm in diameter. If the columns are fixed beds, they range from 1-2 meters high and are connected in series. If a pulse bed is used, the bed depth is usually 10-15 meters. Assuming there are sufficient sample points in both types of systems, the same quality of data can be obtained. The pulse bed has the advantage of easy carbon addition and removal.

Carbon type to be used will usually have been determined during the isotherm testing. Since some carbons used in chemical operations are used on a throwaway basis or can be reactivated to near virgin activity, testing with virgin carbon is representative. Because pore diffusion is a major contributor to the overall diffusion rate, selection of the particle size could be critical. The smaller the particle, the faster the diffusion, and thus, the shorter the mass-transfer zone. This is illustrated in Figure 31 and it is obvious that in a single fixed bed the column having the smaller-diameter carbon particles would treat a greater volume of liquid if it had to be taken offstream at $C/C_o = 20$ percent. However, if the full-scale carbon system is designed to remove only saturated carbon from the system by using beds in series or a pulse bed, the carbon dosage would be the same for either particle diameter carbon. Through the years, there have been many studies conducted and reported as to if and how linear velocity affects diffusion rate.
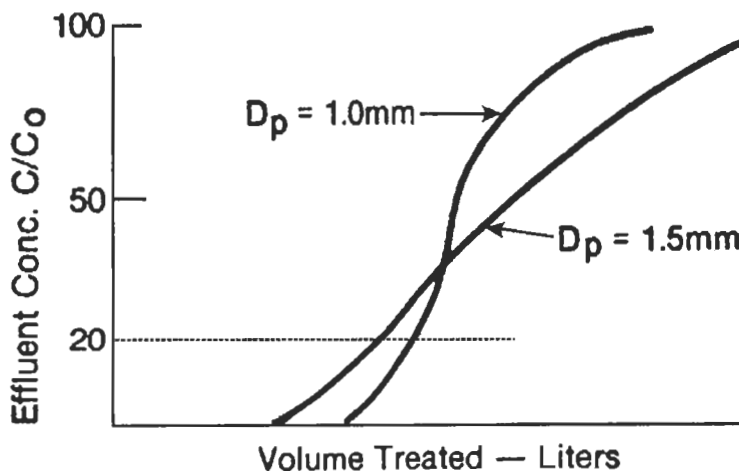
Figure 31. *Effect of particle size on breakthrough curve.*

There are data showing that at the same contact time, but different linear velocities, there is no difference in the performance of a carbon system. It is obvious then that the effect of linear velocity on the diffusion through the film around the particle and the ratio of the magnitude of the film diffusion to the pore diffusion are the factors that determine the effects, if any, that occur. Therefore, the linear velocity cannot be ignored completely when evaluating a system. Systems at the higher linear velocity (LV) treat more liquid per volume of carbon at low-concentration levels and the mass-transfer zone (MTZ) is shorter.

The MTZ is that length of the adsorbent bed where the adsorbate concentration in the liquid varies from the influent concentration to zero or the specification value. At the same contact time, the single fixed-bed system at the higher LV stays onstream about 15 percent longer.

As adsorption is a function of diffusion rate and as diffusion is affected by the liquid viscosity, columns should be operated at the plant process or isotherm temperature to eliminate this variable. If the isotherm investigation indicates that a pH adjustment is necessary before carbon treating, then the same pH adjustment should be made to the liquid before granular carbon column studies. In cases where suspended matter is present in the test liquid, it should be removed by filtration before the liquid is pumped to the columns. If the test liquid has been stored at a low temperature for preservation purposes, subsequent heating to room temperature or higher may result in degassing the liquid. When these conditions exist, the liquid must be deaerated prior to pumping it through the carbon columns. A common practice in adsorption column work which leads to poor adsorption results is that the carbon is not deaerated prior to the adsorption tests. If this is not done properly, air pockets form in the column and result in channeling, high-pressure drops, and premature breakthrough of adsorbate. The time required to deaerate carbon is a function of the liquid temperature.

Carbon should be prewetted prior to being placed in the test columns. Backwashing the carbon at low rates (2.5 m/hr) does not remove the air. Rates that would expand the bed 50 percent or 15-30 m/hr, are required. The liquid used for prewetting can either be water, if it is compatible with the liquid to be treated, or a batch of the liquid to be treated which has been purified previously. There are three types of carbon systems: (1) fixed beds, (2) pulse beds, and (3) fluidized beds, and these can be used singly, in parallel, or in combination. The majority of systems are either fixed or pulse beds. The two basic types of adsorbers which can be designed to operate under pressure or at atmospheric pressure are the moving or pulse bed and the fixed bed. Either can be operated as packed or expanded beds.

In the pulse bed shown in Figure 32, the liquid enters the bottom cone and leaves through the top cone. The flow of liquid is stopped periodically, spent carbon is withdrawn (pulsed) from the bottom, and virgin or reactivated carbon is added into the top of the adsorber. In a fixed-bed adsorber (illustrated in Figure 33), the liquid

passes through the carbon until the carbon is spent or the effluent specification is achieved. The entire volume of carbon is removed from the adsorber and virgin or reactivated carbon is charged to the adsorber.
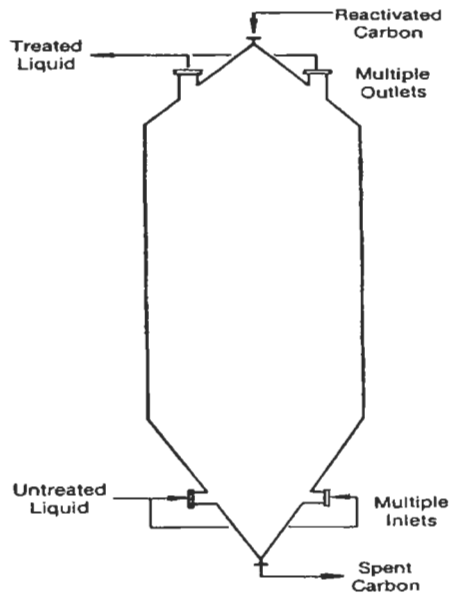
Figure 32. *Moving or pulsed-bed adsorber.*
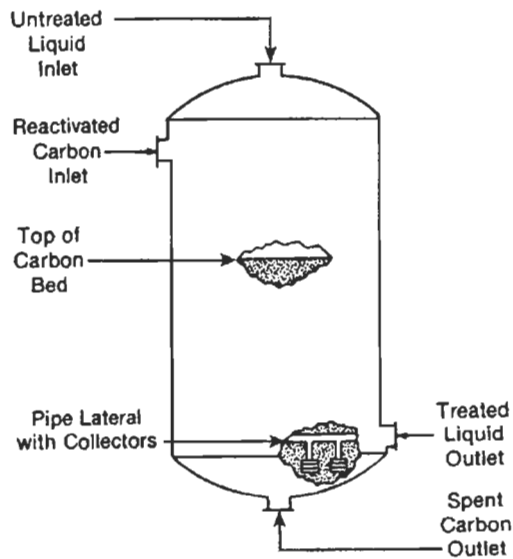
Figure 33. *Fixed-bed adsorber.*

Carbon Regeneration

Carbon systems usually consist of two distinct operations: (1) the contact (adsorption) process, and (2) a carbon regeneration system. A typical arrangement of equipment for use in a granular carbon system is shown in Figure 34. Process liquor is pumped in the column, which is packed with granular carbon and the purified process liquor is removed at the top of the column. Spent carbon is removed from the column periodically for regeneration and then fed back into it. Fresh carbon for makeup is added to the top of the column. The operation of the carbon regeneration furnace can best be described by considering it as part of a regeneration system. All the equipment is directly integrated with the furnace, as shown in Figure 35.
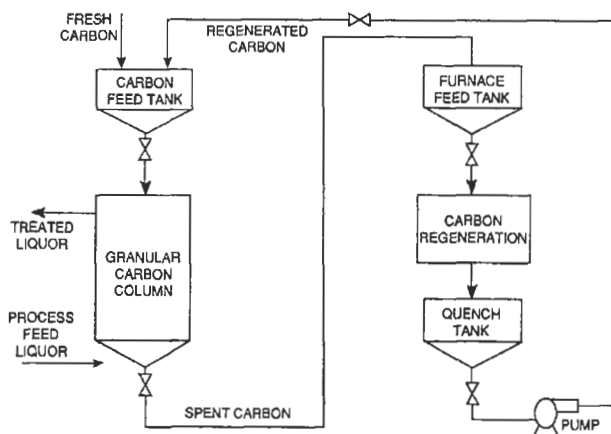
Figure 34. *Equipment arrangement for adsorption system.*
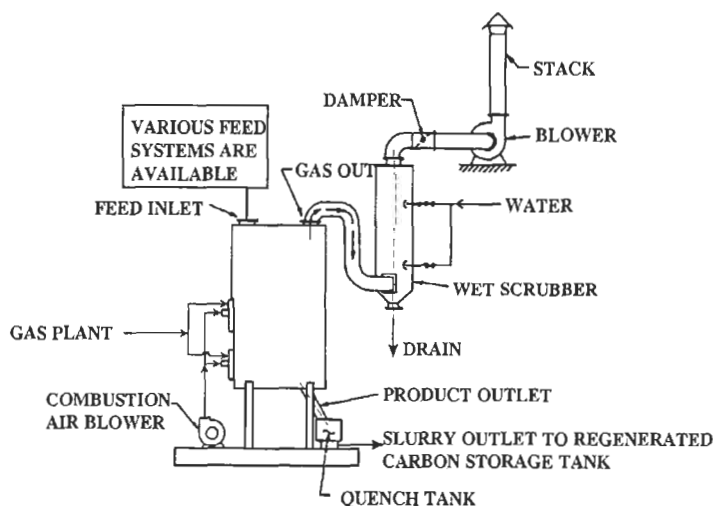
Figure 35. *Carbon regeneration furnace.*

A regeneration system consists of several operations, including conveying of the slurry, receiving, dewatering, feed tank operations, feeding the regeneration furnace (MHF = Multihearth furnace), quench tank and conveying, off-gas equipment operations, and controls and instrumentation. As the carbon is discharged from the carbon columns, it has to be conveyed to a dewatering device before feeding it to the furnace. The carbon is conveyed in a slurry form, which is handled by such equipment as centrifugal pumps, diaphragm pumps, blow cases, and torque flow pumps. The carbon slurry is difficult to convey and care should therefore be taken in the design of the piping. An important consideration is to keep the slurry moving, which will keep the carbon in suspension. To accomplish this, the velocity in the pipes should be above 2.5 ft/sec and preferably above 3.5. Also, elbows should not be used. In their place, long radius bends with a radius equal to at least three times the pipe diameter should be used. Flushing or backwashing connections at certain locations such as at the foot of the risers and other key places have to be considered when laying out the piping. In addition, sight-flow indicators can be installed closer to the pumps or other accessible locations for a visual check that the slurry has not settled and plugged the piping. All valving should be constructed with stainless steel ball valves. If tight shut-off is not required, a butterfly valve may be used. The piping should be kept as short as possible; that is, the regeneration equipment should be placed as close as possible to the carbon columns.

The carbon slurry has to be received and dewatered before feeding it to the furnace. There are two basic carbon column operating systems: the batch and the intermittent or slug type. Depending on which system is used, the receiving, dewatering, and feed operations are performed differently.

In the batch system, several columns or cisterns operate in series, and one extra column is on standby. When a column is operating and then is switched to standby, all the carbon in that column has to be regenerated within a predetermined length of time. The complete column, therefore, has to be transferred to the receiving and/or dewatering tank and then fed to the furnace. As the columns are spent and a new column put-in service, it is placed last in the sequence to have the best carbon in contact with the cleanest water. Dewatering and feeding may be accomplished by one of the following methods:

- On large installations, such as municipal wastewater or sewage water treatment, a single floor-mounted tank, the same capacity as the carbon columns, may be used. It functions as a storage tank to have the carbon column empty to receive the regenerated carbon charge. Another similar tank could also be used as storage for the regenerated carbon. The tank is provided with a shallow cone bottom, an overflow screen, flushing nozzles, and a discharge nozzle. Preferably, it should be covered (refer to Figure 36). The carbon slurry from this tank is discharged into a dewatering screw conveyor.

- On small installations an elevated tank above the furnace would be used (refer to Figure 37). The savings from eliminating the pumps, valving, and slurry lines are offset by the additional cost of a structure to support the tank and a rotary feed valve. The tank is fitted with a screened overflow. A dewatering screw conveyor is used for feeding the furnace.

- Another variation is to use a pyramid-shaped tank at grade level with an inclined dewatering screw feeder. Refer to Figure 38 for an example. Tank materials are usually constructed from carbons steel, stainless steel, stainless clad, resin or epoxy-lined for those situations where iron contamination in the product is a concern, or if corrosion by the adsorbate in the carbon is possible.
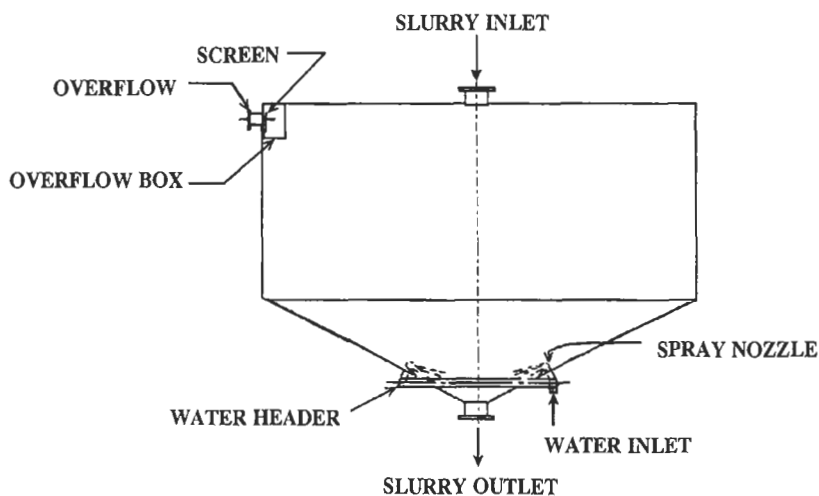
Figure 36. *Storage tank dewatering system.*
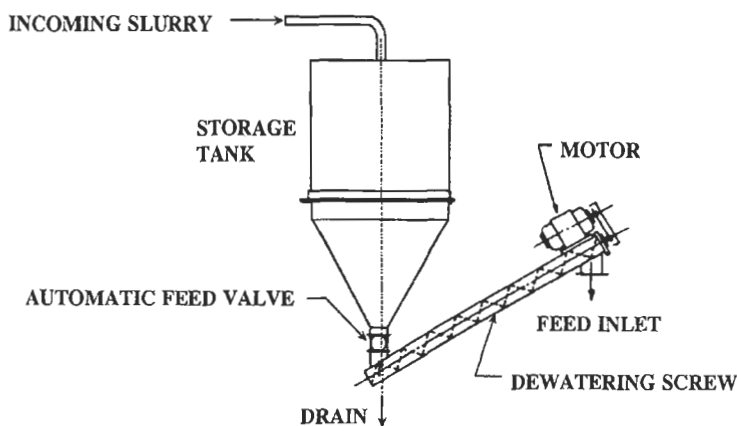
Figure 37. *Storage tank configuration.*
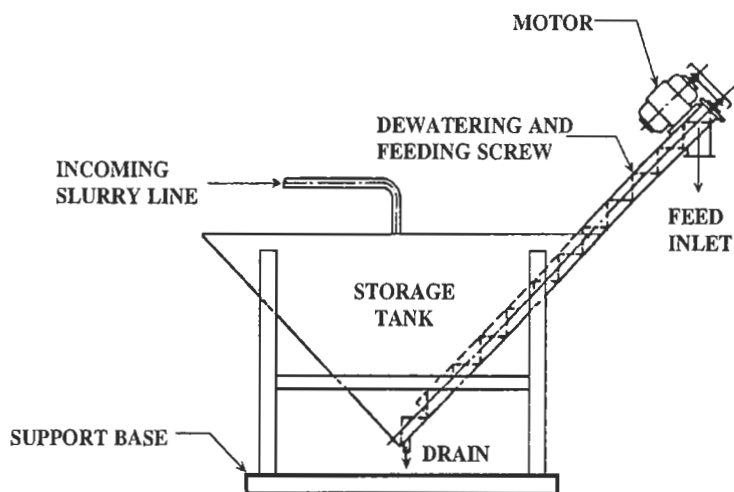
Figure 38. *Example of conical storage tank.*

Intermittent systems are also known as the slug or moving-bed type. A slug of spent carbon is intermittently withdrawn from the carbon column. Flow through the column is only stopped briefly during the time necessary to take the slug out and replenish it with regenerated carbon at the top. Carbon in the column moves countercurrent to the fluid being processed. Figure 38 shows the method used for receiving, dewatering, and storage for a two-tank arrangement. The top tank has a special type of screen fitted to the coned bottom; the water is drained into a special box and out. These tanks have to be fitted with vibrators and also with air backflushing to dislodge the dewatered carbon and discharge it. The screen is a special nonclogging type and is used in carbon installations. It is mounted on a specially made underdrain frame, which is stitch-welded to the tank bottom. This screen is always fabricated of stainless steel. Capacity of the tank should be enough to take in one slug from the carbon column. The slug size is established by experience in the behavior of the carbon in that particular application, which determines the size and frequency of the slugging operation. Underneath the dewatering tank, there is a discharge valve. Normally, this valve is pneumatically operated by remote control from the panel. The control circuit is interlocked with the slurry pump starter so that if the valve is open, the pump cannot be started. In this way, the water from the slurry cannot be dumped into the hot furnace, thereby causing extensive damage to the brickwork. The valve can be with a special ring seat or a positive, closing-type valve. When the valve is opened, the dewatered carbon is dropped from the top tank into the bottom tank, which is called a feed or surge tank. The latter is usually slightly larger in size to ensure that all the carbon can be dropped with no overflow. The materials of construction are carbon steel, stainless steel, stainless steel clad, epoxy lined, and/or resin. The surge tank has a

flanged discharge where the screw feeder is attached. Two other methods are used for receiving, dewatering, and storage. The tank sizes for an intermittent system are smaller since they only have to receive a slug which may be no more than 100 to 200 ft^3 in size.

Feeding equipment can be divided into belt, screw feeder, and rotary valve types. The belt conveyor can be used for feeding but must be wide to fit the discharge. In turn, the tank discharge cannot be reduced beyond certain limits, otherwise bridging will occur. Due to its width, it has to be run extremely slowly as the carbon feed rates are normally very small. Therefore, this requires an intermittent operation to fit the low rates. In addition, a separate seal, usually a rotary air lock, has to be provided at the furnace inlet. Furthermore, wet carbon may stick to the belt, which will result in a dirty operation. The one advantage of the belt is that carbon particles do not undergo deterioration. Screw conveyors can be used for feeding. Several advantages can be realized with this type of equipment. It seals the furnace inlet because by using a covered trough and a flanged connection to the feed tank, the carbon itself serves as the sealing medium, and it eliminates the rotary air lock. It can be slowed down sufficiently to feed the very low feed rates to the furnace and the drive can be of the variable-speed type so the feed rates can be varied at will, and it makes a clean installation. The screw feeder is normally furnished in stainless steel construction, although in some cases, a carbon steel trough can be used. The screw conveyors that can be used on installations include: a plain screw feeder (illustrated in Figure 39), a dewatering inclined conveyor, in which case it is fitted with a dewatering screen at the back end (Figure 37), a dewatering and feeding conveyor (Figure 38).
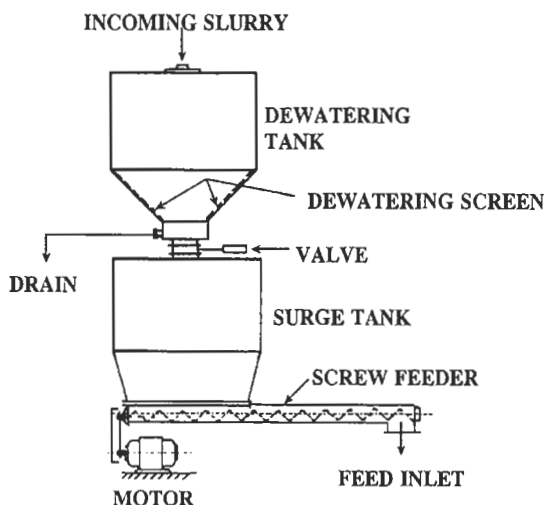
Figure 39. *Surge tank dewatering operation.*
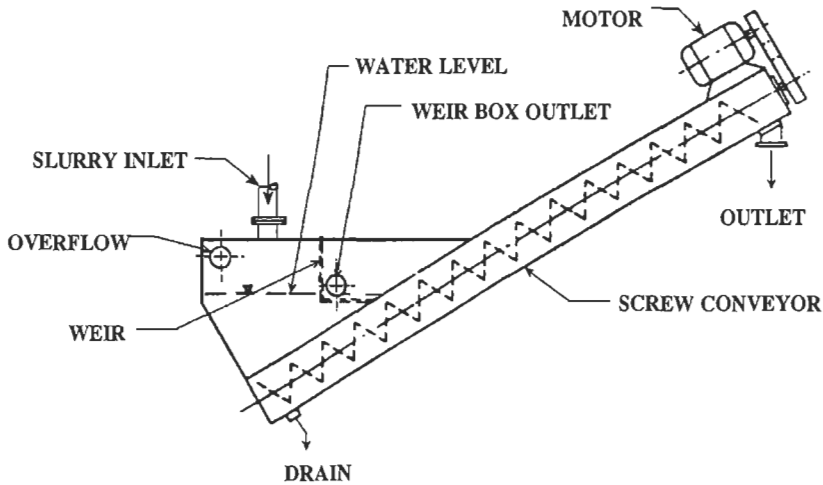
Figure 40. *Inclined screw feeder.*

Another version of the dewatering screw that has been used successfully in a number of installations (Figure 40) consists of an inclined screw with a box-like back end and an internal overflow weir. The drive is usually a variable-speed motor reducer. Rotary air lock fitted with a variable-speed drive is typically used as shown in Figure 37 in connection with the plain receiving tank and an inclined screw conveyor. Materials of construction can be chrome-plated cast iron or nickel-hard casing. The rotary valve is made of hard faced tips or stainless steel replaceable blades.

A multihearth furnace is typically used in the regeneration process. The multihearth furnace employs a simple design approach. It consists of a steel sheet lined with refractory inside. This refractory can be a castable as used in the 30-inch units, or brick as used in the larger sizes. The latter can also have 4.5 inches of insulating blocks which make the walls a total of 9 inches thick for the small furnaces or 13.5 inches on the larger furnaces where high temperatures are used. The interior space of the furnace is divided by horizontal brick arches into separate compartments called hearths. Alternate hearths have holes at the periphery or at the center for the carbon to drop through from one hearth to the next. Through the center of the furnace goes a rotating shaft driven at the bottom by a speed reducer with variable-speed drive. It is scaled at the top and bottom by special sand seals to prevent air or gas leakage. The shaft is hollow and has sockets where arms, called rabble arms, are fitted. An inner tube in each arm and in the shaft provides the means for air cooling of both to prevent damage by the intense heat. This cooling air is blown in through a special connection at the bottom of the shaft. The arms are, in turn, fitted with rabble teeth, placed at an angle, and impart a motion to the carbon when the

shaft is rotated, moving it in some hearths and out in the others. The hearths that move the material out and which have the peripheral holes, called drop holes, are fitted with lute caps. A lute cap is just a disk attached to the shaft which prevents the material dropping from the hearth above. Burners are attached at certain locations in the shell to heat the furnace to the required temperature. These burners can be of the nozzle-mixing or premixed type and are normally set so excess air can also be introduced through them for the oxidation of the organic impurities in the carbon. The burner system includes the usual complement of accessories such as spark-ignited pilots, solenoid valves, mixers, regulators, safety shut-off valves, combustion air blowers, and so on. Fuel used can either be No. 2 diesel oil or gas, either natural, propane, or butane. The shell is also provided with nozzles for injecting steam and air in certain hearths to aid in the regeneration of the carbon. As the carbon is discharged from the furnace, it drops into a quench tank. This is just a small tank filled with water up to a level set by a level controller or float valve. The discharge from the furnace is done through a stainless steel chute, which can either be submerged in the water or fitted with a water curtain. In both cases, isolation of the red hot carbon from the air is obtained. The quench tank can be fabricated from carbon steel, or if iron contamination is objectionable, stainless steel, stainless steel clad, epoxy lining, or resin can be used then. Fines floating in the water are discharged through an overflow to drain. Figure 41 shows a cross-section of a multihearth furnace.
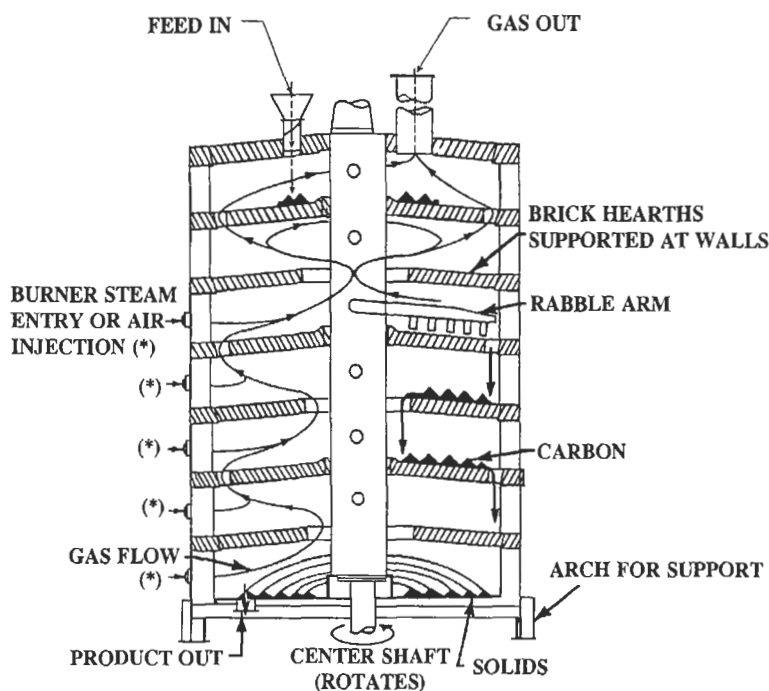
Figure 41. *Cross section of multihearth furnace.*

The multihearth furnace is essentially a device to accomplish heat and mass transfer between gases and solids, passing the gases and solids countercurrently through a series of compartments or stages. In each of these stages, the gas travels in mixed laminar flow over solids spread in thin furrowed layers that are periodically raked to both mix the solids and advance them through the compartment. Heat transfer is both by direct convection and radiation from the gas to the thin widespread area of solids, and by indirect transfer to the brick walls and parallel brick compartment roof followed by re-radiation to the solids, plus minor amounts of conduction through the hearth floor from the gases of the next compartment below. Mass transfer is solely by convection as the gases pass over the solids in laminar flow. Burners, supplying oxidizing or reducing products of combustion of fuel burned, nozzles injecting steam, or air-injection nozzles apart from fuel burners can be placed at any hearth. Thus, temperature and atmosphere can be changed quite sharply from one hearth compartment to the next. If the carbon reaction zone covers two or more hearths, early reaction while the carbon is heavily laden with residue from adsorption may be carried out at one temperature and atmosphere. The final reaction as the carbon nears the regenerated state may be carried out under a different atmosphere or temperature. As to solids flow, it is apparent that the carbon is spread relatively thinly over a large area and stirred periodically. For a given furnace, and at given volumetric feed rate, the faster the center shaft is turned, the shorter the retention time. The conveyance of material through the furnace is by positive displacement. All particles have equal retention time except for some slippage and/or some short-circuiting, giving about a $\pm$ 15 percent range to particle retention time under conditions of normal bed depth. The faster the center shaft is turned, the more frequent the stirring and the shorter the interval in which particles lying on top of the bed remain there. It is usually considered that thermal regeneration of carbon occurs at 1,500 to 1,850 °F by reaction of water vapor and/or carbon dioxide with whatever is left of the adsorbate after it, along with the original carbon, has been heated to these temperatures. Reaction cannot begin until the material has been heated to the reaction temperature, and heating cannot begin until the carbon has been dried. Thus, there are three steps which may, with best design and operation, occur in three separate furnace zones rather than overlap. They are a reaction zone, a heating zone, and a drying zone, considering them from the bottom of the furnace upward, following the path of the gas flow, and also naming them in their probable order of importance rather than their order of occurrence. The reactions expected are:

- $C + H_2O \rightarrow CO + H_2$ (endothermic 4,800 Btu/lb carbon); K equilibrium 1,700 °F = 47.8

- $C + CO_2 \rightarrow 2CO$ (endothermic 5,950 Btu/lb carbon); K equilibrium 1,700 °F = 52.5

- $CO_2 + H_2 \rightarrow CO + H_2O$ (endothermic 37.3 Btu/ft any constituent); K equilibrium 1,700 °F = 0.73

Even at 1,500 °F, equilibrium constants for the first two reactions are high enough (about 10) to expect reaction to go essentially to completion except for kinetic-rate limitations. The reaction zone might be expected to be sized by volume of rabbled carbon bed, considering that the carbon gasification reactions that occur in it are governed by kinetics and are reaction-rate limited. Actually, it is sized by hearth area. The area exposed to the gases controls mass transfer of reactants from the gas phase to the carbon and heat transfer to support the endothermic reactions.

Fluidized-bed powdered activated carbon systems represent another important process. The use of activated carbon for the tertiary treatment of secondary sewage effluents has been used extensively. Powdered carbon is as effective as granular activated carbon for removing the organic impurities from the wastewater.

Before powdered carbon can be used commercially or reused for tertiary treatment of sewage effluents, a method of regeneration is required. The use of the fluidized bed for regeneration offers the key advantages of excellent temperature and atmosphere control and the ability to process the powdered solids conveniently and continuously.

Two other methods worth discussing are wet air oxidation and regeneration by steam. Wet oxidation may be defined as a process in which a substance in aqueous solution or suspension is oxidized by oxygen transferred from a gas phase in intimate contact with the liquid phase. The substance may be organic or inorganic in nature. In this broad definition, both the well known oxidation of ferrous salts to ferric salts by exposure of a solution to air at room temperature and the adsorption of oxygen by alkaline pyrogallol in the classical Orsat gas analysis would be considered wet oxidations.

Most applications of commercial significance require some elevation of temperatures and pressures. A range of about 125 °C (257 °F) and 5 atm. to 320 °C (608 °F) and 200 atm covers most cases. Frequently, air is the oxygen-containing gas, in which case the process may be termed wet-air oxidation (WAO). In the general case, including the use of pure oxygen, the broader term of wet oxidation (WO) is used.

WO may be controlled for a specific synthesis step, held at intermediate degrees in some applications, or forced to completion for waste disposal. Variables that affect wet-oxidation rates are temperature, total pressure, partial pressure of oxygen, gas-liquid phase mixing, concentration of oxidizable substances, pH, and catalysts. The nature of the material will often determine whether a concentration can be altered, a pH adjusted, or a catalyst used. Solubilities of any dissolved solids at the system conditions must also be considered. Another restriction on pressure in many cases is the need for maximum utilization of oxygen. If an excess pressure of oxygen is maintained throughout the liquid-phase detention time, some oxygen will be wasted. The most cost-effective oxygen partial pressure must be considered for each individual case. The overall rate of oxygen transfer from the gas to the liquid phase

is limited primarily by the resistance to mass transfer in the liquid-diffusion layer according to the following equation:

$$r = K\,k_1 a\,(C - C^*)$$

where r is the rate of oxygen transfer per unit volume (kg mol/m^3/sec or lb mol/ft^3 /hr), K is the oxygen solubility constant in water (kg-mol oxygen/m^3 water per kg mol oxygen/m^3 air), k_1 is the liquid film mass-transfer coefficient (m/sec or ft/hr), a is the interfacial area per unit volume (m^{-1} or ft^{-1}), C is the oxygen concentration in the gas phase (kg-mol/m^3 or lb mol/ft^3), and C* is the hypothetical oxygen concentration in the gas phase that would be in equilibrium with the liquid phase (kg-mol/m^3 or lb mol/ft^3). The solubility of oxygen in water, K, increases strongly with temperature and may to some extent be affected by the presence of substances dissolved in water. The liquid film mass-transfer coefficient, k_1, depends on the diffusion coefficient of oxygen in water, the concentration and type of substances dissolved in water, the intensity of stirring in the liquid phase, and the presence of the chemical reaction increasing greatly with increases in temperature, The interfacial area, a, depends on the liquid surface tension, the liquid physical properties, the gas-phase flow rate, the intensity of stirring in the liquid phase, and the concentration and type of substances dissolved in the aqueous phase. The interfacial area also increases greatly with temperature. Because of the difficulty in separately measuring the liquid film mass- transfer coefficient and the interfacial area, these two quantities are most often correlated together as the volumetric mass-transfer coefficient, $k_1 a$. As shown by the equation, the solubility of oxygen in water can have a strong influence on the rate of oxygen transfer from the gas phase to the liquid phase. Solubility increases strongly with temperatures over the range of temperature; above the critical temperature and pressure of a particular air-water mixture, the solubility is considered infinite. Though the solubility of oxygen in water at ambient conditions may be somewhat affected by the presence of dissolved substances, its effect in aqueous solutions at wet-oxidation temperatures and pressures is expected to be very minor when all factors are considered.

Adsorbed solvents and some organics may be stripped from activated carbon by means of direct steam. This method is essentially an in-place regeneration of carbon in the on-line adsorber. The stripping process is a dynamic process and the quantity of steam required to desorb a given amount of carbon is dependent on the physical design of the adsorption plant rather than on the theoretical amount of heat required to distill the solvent from the carbon. Care must be taken with the design of the steam manifold, carbon-bed distillate piping, and condensing system to achieve an efficient steam consumption. A well-designed plant will have a steam consumption in the region of 1 to 4 pounds steam per pound of recovered solvent. During the steaming period, a certain amount of condensation takes place in the carbon bed, increasing the moisture content of the carbon. The amount of moisture may be controlled to a certain extent by the dryness of the steam used and the pressure and temperature conditions in the carbon bed during steaming. In order to achieve the

maximum adsorption capacity and adsorption efficiency, when recovering miscible solvents, it is desirable that the carbon bed should be dried and cooled before being returned to the solvent air stream. The desired state of dryness depends on the physical properties of the solvent and concentration of the solvent in the air or gas stream. It is desirable when using high-solvent concentrations to leave some moisture in the carbon so that the heat of adsorption may be used in evaporating the moisture from the carbon, thus preventing an undue rise in the temperature of the carbon bed. Air should not be used for drying when using solvents which are prone to oxidation, polymerization, or tend to decompose upon contact with the hot, moist activated carbon. The catalytic effect of activated carbon in this condition may cause such solvents to break down or to hydrolyze, resulting in the formation of acids. In such cases it is necessary to dry and cool the carbon with an inert gas. The reader may refer to Section (B) of the reference section of this chapter for selected literature providing more in-depth discussions on adsorption.

SOLVENT EXTRACTION

Principles of a Three Component System

To extract a desired component A from a homogeneous liquid solution, one can introduce another liquid phase which is insoluble with the one containing A. In theory, component A is present in low concentrations, and hence, we have a system consisting of two mutually insoluble carrier solutions between which the solute A is distributed. The solution rich in A is referred to as the *extract phase*, E (usually the solvent layer); the treated solution, lean in A, is called *the raffinate*, R. In practice, there will be some mutual solubility between the two solvents. Following the definitions provided by Henley and Staffin (1963) (see reference Section C), designating two solvents as B and S, the thermodynamic variables for the system are T, P, x_{AE}, x_{BE}, x_{AR}, x_{BR} (where P is system pressure, T is temperature, and the x's denote mole fractions).. The concentration of solvent S is not considered to be a variable at any given temperature, T, and pressure, P. As such, we note the following:

$$x_{AE} + x_{BE} + x_{SE} = 1, \text{ and}$$
$$x_{AR} + x_{BR} + x_{SR} = 1$$

Based on an old principle known as the phase rule, there are three degrees of freedom (F = 3 - 2 + 2). Hence, if at constant T and P, any one variable, say x_{AE}, is designated, all other variables (x_{BE}, x_{AR}, and x_{BR}) are fixed. The phase-equilibrium diagram for a three component system is illustrated in Figure 42. In this figure, the data are plotted on a triangular diagram where the three apexes represent pure *A*, *B*, and *S*. The two-phase region is enclosed within the saturation line, the region to the right of the "plait point" denoting saturated solvent rich (extract) phase, the

region to the left being the saturated raffinate phase. At the plait point the two phases have identical compositions. According to the phase rule, at a fixed T, P, and x_{AE}, the compositions x_{BE}, x_{SE}, x_{AR}, x_{BR}, and x_{SR} are also fixed. This is shown by the tie line **FG** which connects the point xAE (the concentration of A in the extract phase) with x_{AR} (the concentration of component A in the raffinate phase) in equilibrium with x_{AE}. Figure 43 is another type of equilibrium diagram that basically conveys the same information. The coordinates in this second plot are based on a solvent-free basis. The slope of the line connecting xAR and xAE is the ratio of solvent to solute (S/A).
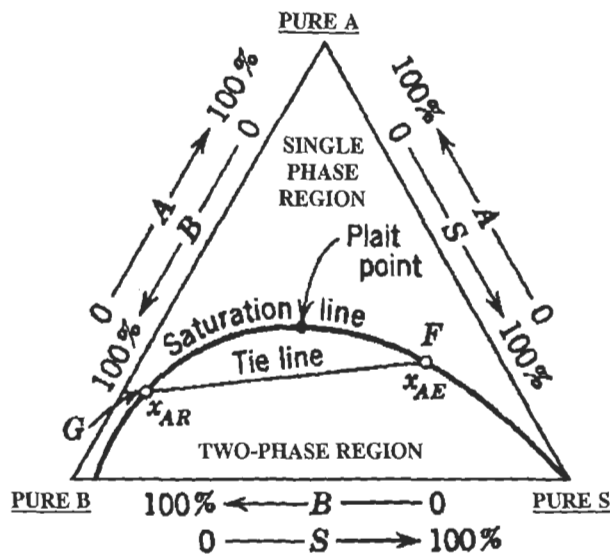
Figure 42. *Phase equilibrium diagram; constant P, T.*
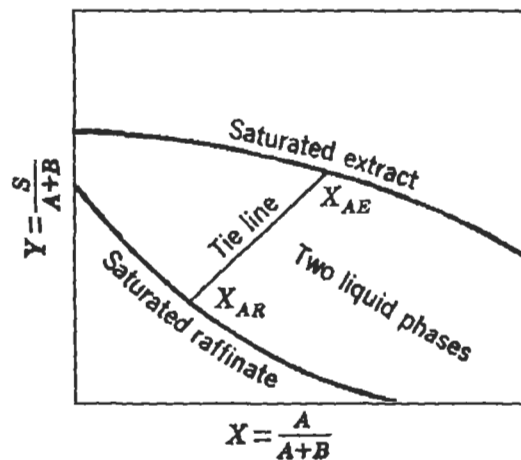
Figure 43. *Equilibrium curve on solvent free basis.*
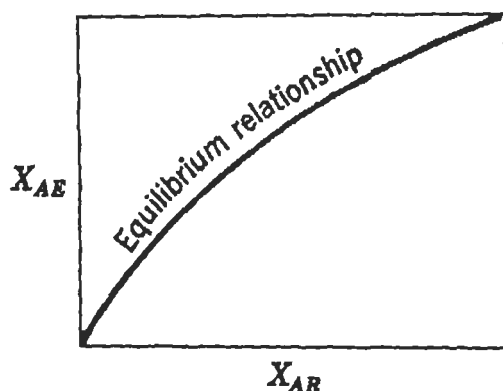
Figure 44. *Idealized equilibrium curve.*

In the extreme case where both solvents B and S are completely insoluble, then there are only four variables, namely, T, P, x_{AE}, and x_{AR}, and the more familiar equilibrium curve illustrated in Figure 44 can be constructed.

The following example taken from the book by Henley and Staffin helps to illustrate the use of a phase equilibrium diagram. In their example, furfural (F) is proposed as a solvent for the recovery of glycol (G) from water (W). This might be appropriate in a water treatment application. From equilibrium solubility and tie line data reported at 25 °C, the furfural-ethylene glycol-water phase equilibrium diagram shown in Figure 45 is constructed. Point F denotes the composition of the mixture containing 45 % glycol, 55 % water, and 0 % furfural. The addition of furfural to the mixture can be followed on the phase equilibrium diagram along the path F → A. At point M the mixture is 50/50 furfural/feed composition. Note that point M is within the two-phase region (refer back to Figure 42 for definition), and hence we may expect that the mixture will physically separate into two phases; namely the extract phase E (which is 28.5 % glycol (G), 6.5 %water (W) and 65.0 % furfural (F)), and raffinate R (8 % G, 84 % W, 8 % F). By means of a material balance, one can determine the amounts of raffinate and extract phases:

Overall material balance:

$$R + E = X$$

Furfural balance:

$$0.65 + 0.08R = Y$$

Graphical methods at best are simply illustrative for the student today, but they are occasionally referenced by the process engineer. Extraction, like distillation can be viewed as a stage-wise operation, and hence methods based on the McCabe Thiele approach briefly described in Chapter 4 have been applied to preliminary design cases. Indeed, both absorption and adsorption are stage-wise operations.
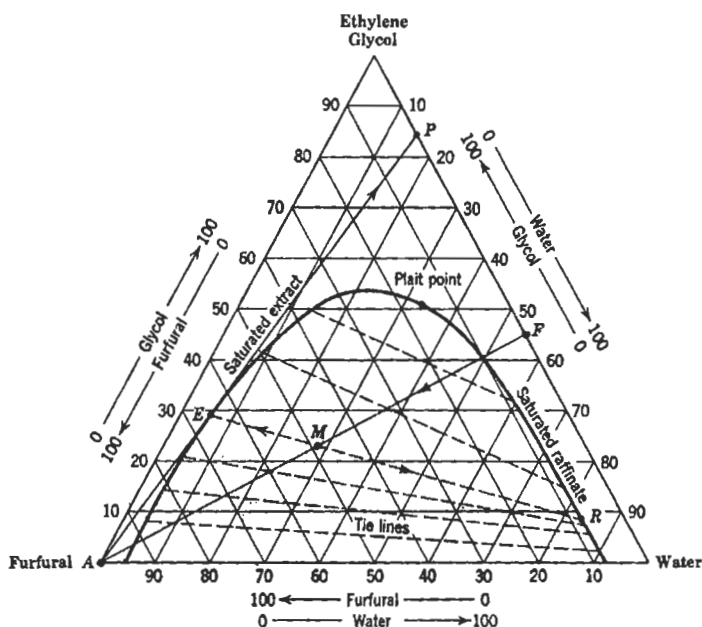
Figure 45. *Furfural-ethylene glycol-water phase equilibrium diagram.*

Practical Considerations

As defined early on, liquid-liquid, or solvent extraction is defined as the separation of the components of a liquid solution by treatment with an immiscible solvent in which one or more of the components of the solution are soluble. In many cases distillation is cheaper and more effective than extraction, but it is also true that separation by the latter method is sometimes practicable where ordinary distillation is uneconomical or actually impossible. The example of acetic acid removal from a dilute solution of acetic acid in water by distillation or by extraction with toluene, in which the acid is soluble and the water insoluble is a classic example. In contrast, close-boiling petroleum fractions, differing chemically, may be almost impossible to separate by distillation but easily separable by solvent extraction. hence, extraction is a preferred method in applications involving the separation of compounds differing in chemical type, but which may be hard to separate by distillation because their volatilities do not differ significantly. The basic process steps are (a) bringing solvent and solution into intimate contact, separation of the resulting phases, and finally, the removal and recovery of solvent from each phase, which can be accomplished by distillation. Contacting the feed and solvent may be accomplished in any of the several types of equipment, such as baffle-plate mixers, impinging jets of the two liquid streams, agitated vessels containing the liquids, plate columns, or even in packed towers. Separation may be accomplished by simple settling tanks or by means of centrifugation. The difficulty encountered in

separating the phases is usually greatest when the phases are dispersed to a high degree in the contacting equipment. A large difference between the densities of the two phases tends to make separation relatively straightforward, but the presence of emulsifying agents may cause more trouble in the separation process than a small density difference. After separation of the phases, the solvent is usually recovered by ordinary distillation of the solvent layer.

In a once-through or single contact process, the solvent and solution are brought together for a batch extraction. The solute is transferred from one phase to the other, and the concentrations in the two phases are allowed to approach equilibrium. Hence residence time is an important design consideration. The amount of solute extracted is governed by the amount of solvent used. Once-through systems are the simplest in design, with the contacting equipment consisting of a variety of types, such as mixers of the jet or agitator types. The contact between phases may be so good that equilibrium is approached closely. The final extract and raffinate from a single-contact operation are essentially in. equilibrium with each other, and the amount of solute which may be extracted using a specified amount of solvent is definitely limited. Because the efficiency permitted by the equilibrium involved is usually low, this type of operation is seldom employed for large-scale operations, but from time to time there are specialty cases. More common is multiple-contact extraction, which uses fresh solvent in each contacting moment. In this case the extraction being repeated on the raffinate from the first contact. The reduction of solute content of the raffinate may be improved to any desired extent by increasing the number of contacts, or specifically, the number stages. The concentration of solute from the second and later separators is low, however, and this method of operation is uneconomical of solvent. Theoretically, the maximum efficiency is achievable when the total solvent to be used is divided in equal parts, with the same quantity being used in each successive stage. Thus if one volume of solution is to be extracted with six volumes of solvent, the best results are obtained by using three volumes of solvent in each mixer stage if the operation is two-stage, two volumes in each mixer if three-stages, or one volume in each mixer if six-stages.

Suitable organic solvents, such as ether, benzene, naphtha and the like, are more soluble than in water. This makes it possible to separate them from other substances which may accompany them in the water solution but which are not soluble in the solvents employed. Hence, one application of solvent extraction is the analytical determination of unsaponifiable oils and waxes in admixture with fatty material by submitting the mixture to vigorous saponification with alcoholic potash or, if necessary, sodium ethylate, and to dilute the product with water and extract with petroleum ether. The soaps remain in the aqueous solution while the unsaponifiable oils and waxes dissolved in the ether. The addition of a salt to an aqueous solution prior to extraction is sometimes practiced in some processes. In older processes, SO_2 is employed in the separation of aromatic and highly saturated hydrocarbons, taking advantage of the much greater solubility of the solubility of the aromatics and

unsatureds in SO_2. This techniques is largely an old laboratory method. Many applications for solvent extraction can often be described as specialty in nature. Where processes have been most economical on the industrial scale has been by countercurrent multiple contacting. Figure 46 illustrates the generalized process scheme. The process schematic shows three stages, although any number may be used. The solution to be treated enters the first contact device, and all of the fresh solvent enters the last contactor. The two streams pass in a countercurrent direction, the original solution being treated with extract from the second stage, and so on, the final extract being removed from the first separator and the final raffinate from the last separator. All of the solvent passes through each stage, and the overall efficiency, for a given amount of solvent and number of stages, is appreciably better than when part of the fresh solvent is used in each stage. This type of operation, with from two to six stages, is the one most commonly employed for commercial solvent extraction. The operation may be either batch or continuous, as exemplified by systems of batch mixers and settlers on the one hand, or by the bubble-cap plate column on the other. We can conclude that the maximum extraction efficiency in any process would be obtained if it were possible to obtain intimate contact between phases in an apparatus in which the two liquid phases passed continuously in opposite directions. Ideal operation are difficult to achieve in practice because the best contact is obtained by violent mixing of the phases. Packed towers have been employed, however, the heavier liquid flowing downward and the lighter liquid passing upward means that the liquids cannot be pumped past each other cannot be achieved without great energy sacrifice., especially when there is a significant difference in density of the two phases. The intimacy of contact obtained in jet mixers is possible in some applications, with the equipment being simple and reasonably cheap. The theoretical maximum efficiency of this type of operation is higher than in the case of the countercurrent batch systems. In the case of batch extractions employing jet- or agitator-type mixers, the contact is so good that equilibrium between phases is closely approached, and the rate of interphase diffusion does not govern performance.
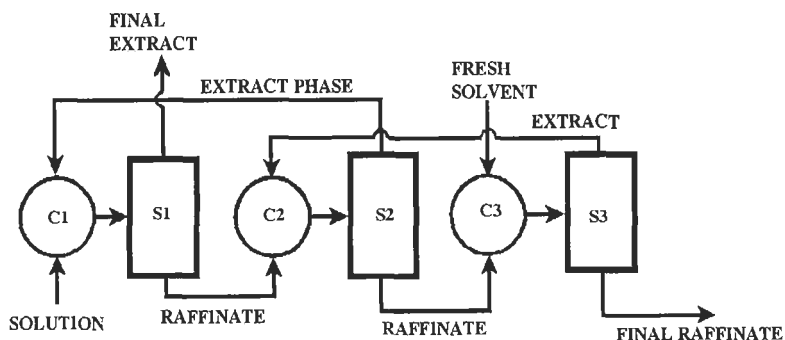
Figure 46. *Simplified schematic of countercurrent extraction process; C-contactor, S- separator.*

The efficiencies which may be obtained can consequently be calculated by simple stoichiometry from the equilibrium data. In the case of countercurrent-packed columns, the solute can theoretically be completely extracted, but equilibrium is not always reached because of the poorer contact between the phases. The rate of solute transfer between phases governs the operation, and the analytical treatment of the performance of such equipment follows closely the methods employed for gas absorption. In the case of two immiscible liquids, the equilibrium concentrations of a third component in each of the two phases are ordinarily related as follows:

$$y = K'x$$

where y is the solute concentration, in one liquid (extract) phase, x is the solute concentration, in the other (raffinate) phase, and K' is known as the distribution coefficient." Equilibrium data for such systems are usually reported in terms of the distribution coefficients, which are analogous to Henry's law constants. For most systems, however, K' varies considerably with concentration. In cases where the solute is associated in one or the other phase, forming double or triple molecules, the mass action and equilibrium laws may be combined to give the equilibrium relation.

Two additional terms of importance are *leaching* and *washing*. Leaching of a solid is a special case of extraction which involves the diffusion of the solute through the liquid film onto the solid surface. Predicting the leaching rate is complex since the interfacial area decreases as the leaching process continues. The term washing is used to describe the process of extraction of a solute already in solution in a liquid entrapped by porous solids or sludge. The reader may refer to some of the references in Section A of this chapter for additional information on these processes.

REVERSE OSMOSIS

Reverse osmosis (RO) for water and wastewater treatment is a well established process operation. Typical applications include desalination for drinking water supplies, removal of salts and minerals for water recycling in power generating plants, preparation of high quality water supplies for boiler makeup, preparation of high quality drinking water in the manufacture of spirits, treating wet sulfur dioxide scrubber waste. The use of RO for desalination of seawater for boiler makeup is a typical application in parts of the Middle East. The availability of this system has opened up the use of heretofore unavailable water supplies, and it has been used by the industry as a pretreatment to ion exchange demineralization. RO acts as an economical roughing demineralizer, bringing down the overall cost and improving the life of resins and operation of the ion exchange equipment. Osmosis is the spontaneous passage of a liquid from a dilute to a more concentrated solution across an ideal semipermeable membrane that allows passage of the solvent (water) but not

the dissolved solids (solutes) as shown in Figure 47. If an external force is executed on the more concentrated solution, the equilibrium is disturbed and the flow of solvent is reversed. This phenomenon, RO, is depicted in Figure 48. A basic RO treatment system consists of the components illustrated in Figure 49. Feedwater to the RO system is pumped first through a micrometer filter. This is a replaceable-cartridge element filter. The purpose of this filter is to remove any turbidity and particulate matter from the feed water before it enters the RO system.
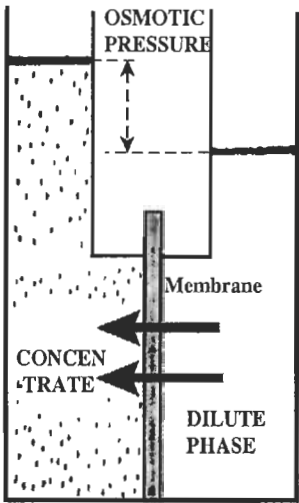
Figure 47. *Reverse osmosis.*
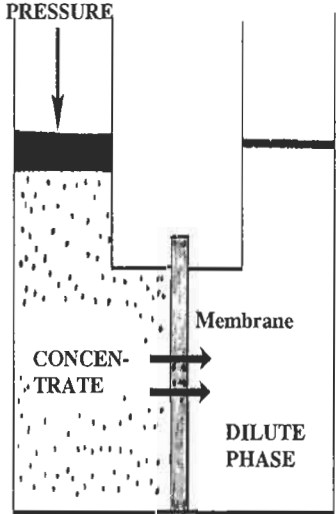
Figure 48. *Flow reversal principle.*
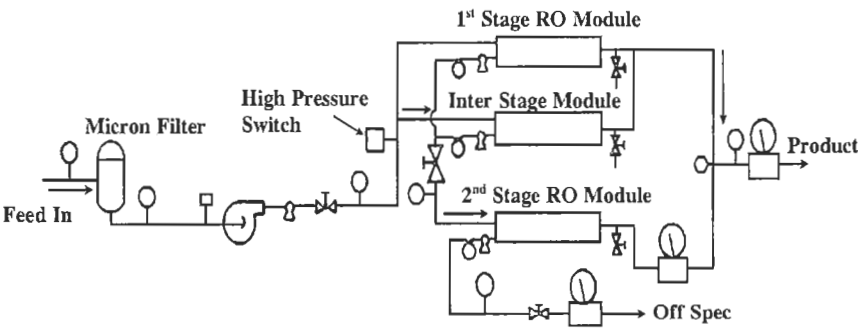
Figure 49. *Basic components of a RO system.*

The filtered raw water then flows to a high-pressure pump, which feeds the raw water at a typical pressure of 400 psi through the RO membrane system. Valves and pressure gauges between the micrometer filter, the high-pressure pump and membrane modules control the flow of water through the system and monitor its operation. The RO system consists of two stages. The raw water is pumped through the first stage, which contains twice the number of membrane modules as the second stage.

The first stage purifies 50% of the water fed to the system and rejects the remaining 50%, which contains all contaminants. This reject water from the first stage is then passed through the second stage, which purifies 50% of the water fed to it and rejects the remaining 50% to waste. This second stage reject (or off-spec) now contains all of the contaminants removed by both stages. Thus, the total flow through the system is 75 % purified product water and 25 % reject water.

The RO system removes 90-95 % of the dissolved solids in the raw water, together with suspended matter (including colloidal and organic materials). The exact percent of product purity, product recovery and reject water depends on the amount of dissolved solids in the feedwater and the temperature at which the system operates.

RO membrane performance in the utility industry is a function of two major factors: the membrane material and the configuration of the membrane module. Most utility applications use either spiral-wound or hollow-fiber elements. Hollow-fiber elements are particularly prone to fouling and, once fouled, are hard to clean. Thus, applications that employ these fibers require a great deal of pretreatment to remove all suspended and colloidal material in the feed stream. Spiral-wound modules (refer to Figure 50), due to their relative resistance to fouling, have a broader range of applications. A major advantage of the hollow-fiber modules, however, is the fact that they can pack 5000 ft^2 of surface area in a 1 ft^3 volume, while a spiral wound module can only contain 300 ft^2/ft^3.

The hollow fine fiber configuration (refer to Figure 51) consists of a bundle of porous hollow fine fibers. These fibers are externally coated with the actual membrane and form the support structure for it. Both ends of each fiber are set in a single epoxy tube sheet, which includes an O-ring seal to match the inside diameter of the pressure vessel.

Influent water enters one end of the pressure vessel and is evenly distributed along the length of the vessel by a concentric distributor tube. As the water migrates out radially, some of it permeates the fibers and exits the pressure vessel via the tube sheet on the opposite end. The direction of permeate flow is from outside to inside the fibers. The concentrated solution, or reject, completes its radial flow path and leaves the vessel at the same end at which it entered.

Figure 52 is a representation of a complete module. For clarity, the vessel and inlet distributor have been omitted. The actual outside diameters of individual fibers range from 3 to 10 mils, depending on manufacturer.
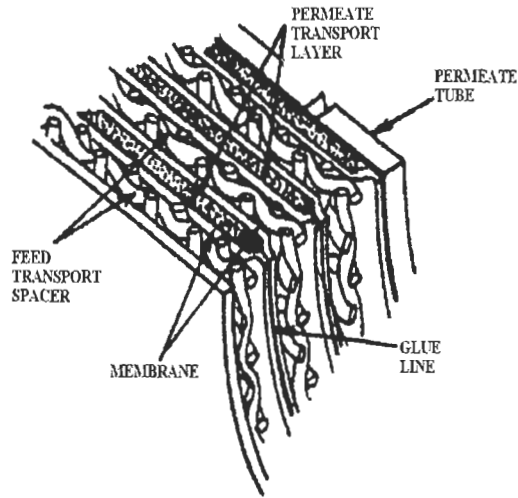
Figure 50. *Spiral wound membrane.*
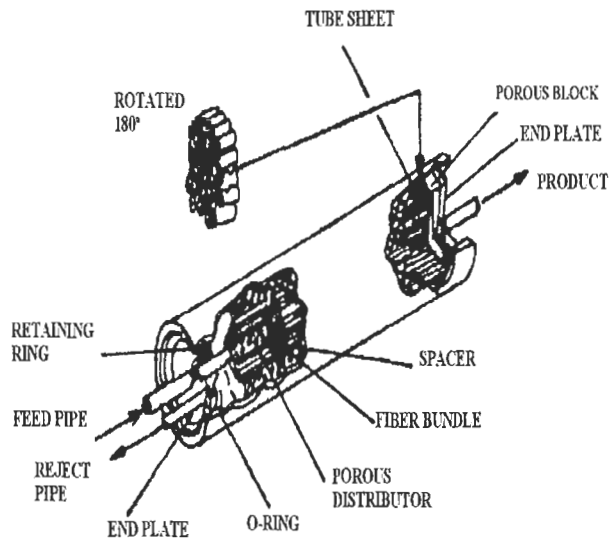
Figure 51. *Hollow fiber module.*

Basically, the spiral-wound configuration consists of a jelly roll-like arrangement of feed transport material, permeate transport material and membrane material. At the heart of the wall is a perforated permeate collector tube. Several rolls are usually placed end to end in a long pressure vessel.
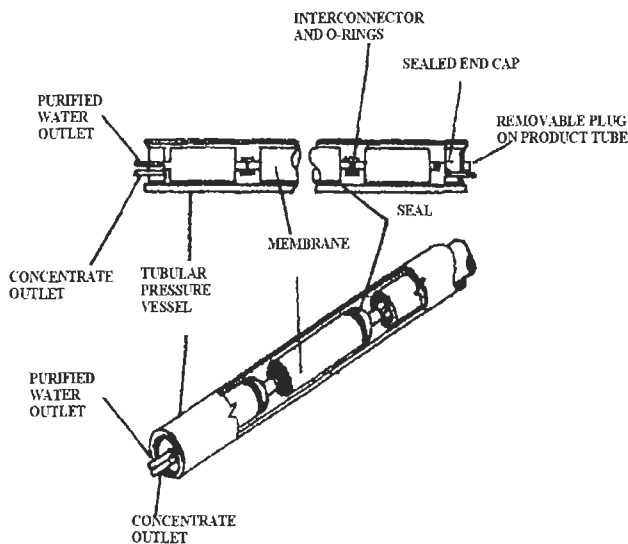
Figure 52. *RO module assembly.*

Influent water enters one end of the pressure vessel and travels longitudinally down the length of the vessel in the feed transport layer. Direct entry into the permeate transport layer is precluded by sealing this layer at each end of the roll. As the water travels in a longitudinal direction, some of it passes in radially through the membrane into the permeate transport layer. Once in the transport layer, the purified water flows spirally into the center collection tube and exits the vessel at each end. The concentrated feed continues along the feed transport material and exits the vessel on the opposite end from which it entered.

Two common types of membrane materials used are cellulose acetate and aromatic polyamide membranes. Cellulose acetate membrane performance is particularly susceptible to annealing temperature, with lower flux and higher rejection rates at higher temperatures. Such membranes are prone to hydrolysis at extreme pH, are subject to compaction at operating pressures, and are sensitive to free chlorine above 1.0 ppm. These membranes generally have a useful life of 2 to 3 years. Aromatic polyamide membranes are prone to compaction. These fibers are more resistant to hydrolysis than are cellulose acetate membranes.

SUGGESTED READINGS

Section A: The following references are recommended for more in-depth readings on absorption and solvent extraction theory, design principles, and practical applications.

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2. Cheremisinoff, N. P. *Chemical Engineer's Condensed Encyclopedia of Process Equipment*, Gulf Publishers, Houston, TX (2000).

3. Danletson, John A., *Air Pollution Engineering Manual*, U.S. Department of Health, Education & Welfare, Cincinnati, Ohio, (1967), pages 210-222.

4. Cheremisinoff, Paul N. and Young, R. A., *Air Pollution Control and Design Handbook, Part 2*, Marcel Dekker, Inc., New York, New York (1977), pages 609-720.

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14. Process for Recovery of Natural Gas Liquids From a Sweetened Natural Gas Stream, U.S. Patent 4,421,535, Dec. 20, 1983.

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16. Hydrocarbon Separation with a Physical Solvent, U.S. Patent 4,578,094, Mar. 25, 1986.

17. Process for Using Preferential Physical Solvents for Selective Processing of Hydrocarbon Gas Streams, U.S. Patent 4,617,038, Oct. 14, 1986.

18. Using extraction to treat hydrocarbon gases, *Chemical Engineering,* Oct. 27, 1986, p. 53.

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20. Extractive Stripping of Inert-Rich Hydrocarbon Gases with a Preferential Physical Solvent, U.S. Patent 4,680,042, Jul. 14, 1987.

21. Process for Using Alkyl Substituted C8-C10 Aromatic Hydrocarbons as Preferential Physical Solvents for Selective Processing of Hydrocarbon Gas Streams, U.S. Patent 4,692,179, Sep. 8, 1987.

22. Conversion of Lean Oil Absorption Process to Extraction Process for Conditioning Natural Gas, U.S. Patent 4,696,688, Sep. 29, 1987.

23. Process for Extractive-Stripping of Lean Hydrocarbon Gas Streams at High Pressure with a Preferential Physical Solvent, **U.S. Patent 4,695,672,** Sep. 22, 1987.

24. Processing Nitrogen-Rich, Hydrogen-Rich, and Olefin-Rich Gases with Physical Solvents, **U.S. Patent 4,832,718,** May 23, 1989.

25. Processing Nitrogen-Rich Gases with Physical Solvents, **U.S. Patent 4,883,514,** Nov. 28, 1989.

26. Processing Hydrocarbon Gases with Selected Physical Solvents, **U.S. Patent 4,883,515,** Nov. 28, 1989.

27. Using Non-Cryogenic Absorption to Reject Nitrogen From Subquality Natural Gases - The Mehra Process[sm] Alternative, *Gas Separation International Conference,* Austin, TX, Apr. 22 - 24, 1991.

28. Non-Cryogenic Absorption-Based Mehra Process[sm] Technology Upgrades Hugoton Gases, *Advances in Nitrogen Rejection Technology Symposium,* **AIChE** *1993 Spring National Meeting*, Houston, TX, Mar. 29 - Apr. 1, 1993, Paper 45(a).

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32. Gas Phase Olefin Polymerization Process with Recovery of Monomers from Reactor Vent Gas by Absorption, **U.S. Patent 5,521,264,** May 28, 1996.

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36. Absorption Process for Rejection of Reactor Byproducts & Recovery of Monomers from Waste Gas Streams in Olefin Polymerization Process, **U.S. Patent 5,681,908,** Oct. 28. 1997.

37. Absorption Process with Solvent Pre-Saturation, **U.S. Patent 5,687,584** Nov, 18, 1997.

38. Guidelines offered for choosing cryogenics or absorption for gas processing, *Oil & Gas Journal,* Mar. 1, 1999, Vol 97, No. 99

Section B: The following references and Web sites are recommended for more in-depth information on adsorption technology and engineering principles. The Web sites are briefly described for the reader below.

1. Cheremisinoff, N. P. and P. N. Cheremisinoff, *Carbon Adsorption for Pollution Control*, Prentice Hall, Englewood Cliffs, New Jersey, 1993.

2. *Activated Carbon in the Marine Tank*: Describes the pro's and con's of activated carbon in marine aquaria. http://www.hallman.org/filter/gac.html

3. *Selective Adsorption Associates Inc.*: Specialists in adsorption and filtration products offering assistance in client to product applications. http://www.selectiveadsorption.com

4. *Barneby & Sutcliffe*: Site describes company providing activated carbon, air purification, solvent recovery, emission control, remediation, odor control, corrosive gas control, pollution control, air filterr, and distillation equipment. http://www.bscarbons.com

5. *Charcoal*: Encyclopedia definitions and general information on adsorption. http://www.encyclopedia,com

6. *NSF International Certification Criteria for Drinking Water Treatment Units*: Various papers on adsorption and other technologies for drinking and wastewater applications. http://nsf.org/consumer/dwtuconsumer.html

Section C: The following Web sites are recommended for additional information on reverse osmosis and membrane technologies.

1. *ZENON Environmental Inc.*: Contains information on membrane technology and equipment descriptions and support services for drinking water treatment applications. http://www.zenonenv.com/zenon_drinking_water.html

2. *Hydrocomponents & Technologies, Inc.*: Site describes products supplied for commercial, industrial and residential applications; membrane production equipment; components for manufacturers of reverse osmosis systems. http://www.hcti.com

3. *Croal and Associates*: Suppliers of filtration equipment and filter media including membranes, reverse osmosis and desalination systems. http://croalassociates.com

4. Cordonna Associates, inc.: Site provides online shopping for reverse osmosis systems. http://caitechnologies.com/water.htm

5. Freshwater 2000: Desalination equipment incorporating new technology which is claimed to operate more economically than conventional systems. http://www.freshwater2000.com

Chapter 6

MECHANICAL
SEPARATION
EQUIPMENT

INTRODUCTION

There are several types of equipment used for either processing intermediate products, product separation or recovery, for improving feed and product stream qualities and purities, and for pollution control purposes - that separate phases making use of the physical properties differences between chemical components. These equipment categories may be used to separate solids or particulate matter from liquids, slurries and gases, to concentrate solids by removing moisture through means other than by the direct application of thermal energy, to segregate solid particulate matter by size or density differences, to separate liquids by means other than solvent extraction or distillation. These equipment are broadly classified as mechanical devices or physical separation equipment. They capitalize on properties such as density, viscosity, surface tension, electrostatic forces, size, and several other properties, whereby the differences between the components, and in some cases the phases of components are significant. These property differences then are the driving forces which form the intended applications and design basis for such equipment. The equipment is so generalized in application that they represent basic unit operations and standard pieces of equipment that are relied on in almost any industry sector. Therefore, specific examples are illustrative only, simply because these types of machinery are versatile in almost any industry sector. This chapter covers the following equipment categories: filtration, settling, floatation and clarification, centrifugation, particle segregation and classification. Emphasis in this chapter is given towards providing discussions on fundamental operating principles and equipment specification criteria. It is an overview chapter, meaning that theory and heavy design practices are not addressed, but rather, sufficient information and additional references, including vendor contacts and Web sites are provided for establishing preliminary equipment specifications. The vast majority of these equipment have been in use in various allied industry applications for more than a century, and hence design criteria are well established based on many tried and proven prototypes and industry applications. Many of the operations and equipment described are often applied in either preliminary, intermediate or

final stages of a process operation, and less often as the primary process step. Classical references sited at the end of this chapter can be consulted for theoretical considerations.

FILTRATION EQUIPMENT

Gas Cleaning Equipment

Fabric filters, more commonly called baghouses or dust collectors, have been in use since the early 1900's in the mining industry. Today's applications extend throughout the chemical process industries with considerable emphasis in industrial air pollution control. Dry dust filters are available in sizes ranging from a few square feet up to several hundred thousand square feet of cloth. Gas flows that can be handled by individual units range from under 100 cfm to over 1,000,000 cfm. The fabric filter's design is similar to that of a large vacuum cleaner. It consists of bags of various shapes constructed from a porous fabric. Filter bags are available in two major configurations, namely, flat (envelope) bags and round (tubular) bags. Figure 1 illustrates the operation of a baghouse. The dust-laden gas enters the module through an inlet diffuser that breaks up the gas stream and evenly disperses the dust. The heavier dust particles settle into the hopper and the fine particles rise through the tube sheet into the bags.
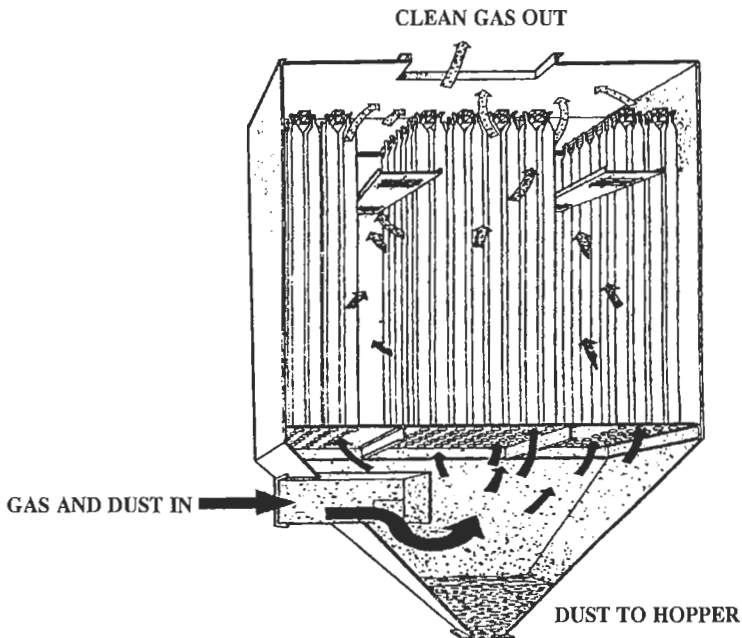
Figure 1. *Basic operation of a baghouse.*
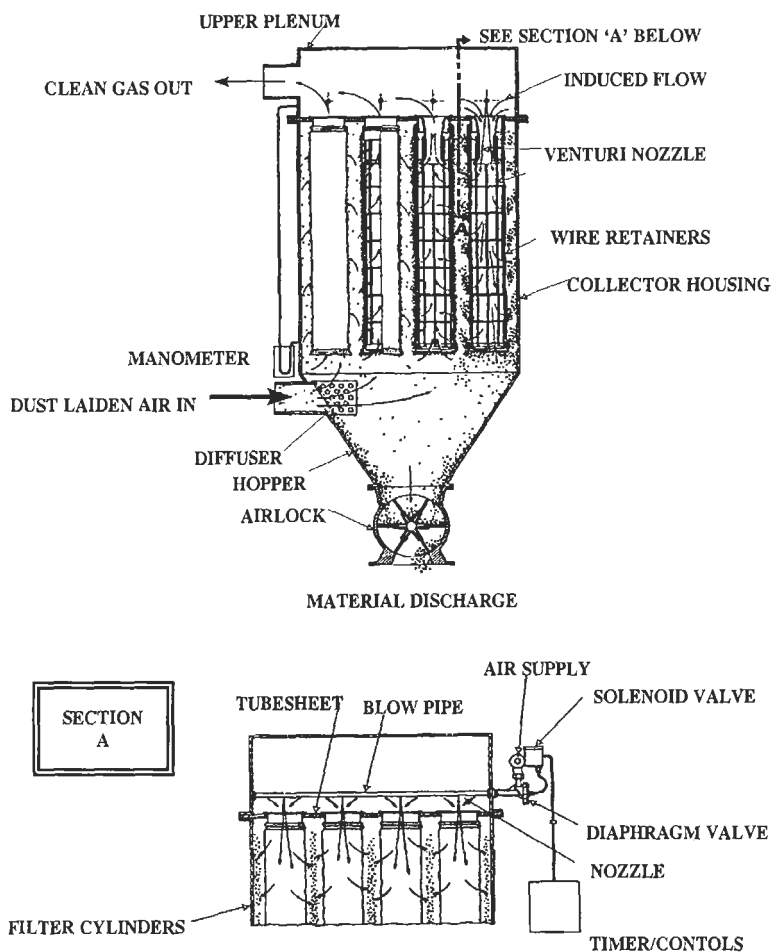
Figure 2. *Design features of a baghouse.*

Particles typically as small as 0.5 microns in diameter are collected on the inside of the bags, while the cleaned gas passes through the fabric. Dust is removed from the bags by periodic shaking accomplished by an automatic shaker. The frequency of cleaning depends on the type of dust, the concentration, and the pressure drop which must be overcome. The dust shaken from the bags falls into the hopper below and is removed by a rotary airlock, screw conveyor, or other devices. Figure 2 illustrates some of the specific design features and components of a baghouse operation. Series modules can be joined to provide any desired capacity. When two or more modules are joined together, a single module can be shut down for bag cleaning and then returned to service. The simple closing of an inlet or outlet damper diverts the dirty gas stream to other modules. Thus, the gas is filtered continuously. Multi-module installations typically employ a large single fan or small

individual fans mounted on each module. Small fan arrangements are more flexible and eliminate the need for outlet ductwork and the foundation that are required for a large fan. Individual fans often simplify maintenance and permit fan, motor, drive, or other components to be changed readily without interrupting normal service. Any one module can be shut down and isolated from the rest of the system while still maintaining full operation and efficiency levels. The particles to be removed play an important role in the selection of a fabric and filter efficiency. Specifically, particle density, concentration, velocity, and size are important. Each of these properties is interrelated to the pressure drop of the system, which has a direct impact on operating or particle capturing efficiency. Principal variables directly related to pressure drop are gas velocity, the cake resistance coefficient, the weight of cake per unit area, and the air-to-cloth ratio. The cake resistance coefficient is dependent on the particle size and shape, range of the particle sizes, and humidity. Weight of the cake per unit area is related to the concentration of particulate matter.

Large scale air filtration systems for air pollution control applications are generally packaged systems. Commercially available packages are available which include both the reverse air and compressed air types. Reverse air baghouses have typical diameters that range from 8 to 18 ft, from 6,000 cfm to 94,000 cfm. Compressed air baghouses usually include both bottom bag removal (i.e., through bin vents) as well as top bag removal units. Commercial systems can be round or square/rectangular in configuration. Round (plan view) body sections are usually retrofitted with square plenum sections.
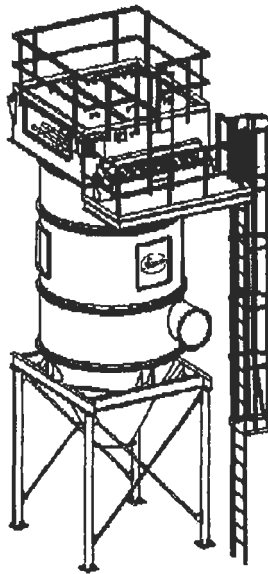
Figure 3. *Round design.*   **Figure 4.** *Square design.*
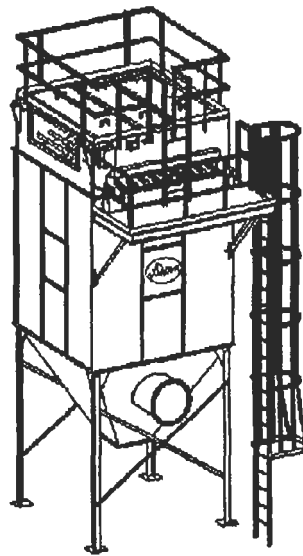
Depending on the accessibility afforded by adjacent equipment, aged ladders and access platforms are often quoted as options by vendors. Square body configuration systems (plan view) have square body and plenum sections. Safety handrails surrounding the plenum are standard. Structural supports are usually optional, the height of which depends on system location. Figures 3 and 4 provide sketches of the two basic configurations.

The design of a fabric filter system must satisfy two criteria, namely, high partcile capture efficiency and low pressure drop. Prediction of the pressure drop and knowledge of its dependence on operating conditions of the filter are necessary for proper design. Fabric filters composed of thicker felt materials have a complex orientation of fibers which can achieve a high collection efficiency with less dust buildup. Consequently, felt filters cannot be cleaned by mechanical shaking due to embedment of fine particles. Instead, a high pressure air stream (60 to 120 psi) is used to disengage the dust particles. The specific application dictates the type of fiber. For example, fiberglass filters can withstand higher temperatures than wool; nylon is a poor fiber to use for chemical resistance. The fabric is designed to withstand thermal, chemical, and mechanical action.

Fabric filters can be categorized according to the particular cleaning method, the filter capacity, the type of filter media, the temperature capability, and the type of service (either intermittent or continuous). There are three major cleaning methods employed: shakers, reverse air, and pulse jet. The oldest and most widely used cleaning method is mechanical shaking. The casing is divided into an upper and lower portion by a tube sheet. The woven fabric tubular bags are located in the upper portion with a pyramid-shape hopper in the lower end. Each bag is supported between a flexible cap and a fixed thimble. Gas velocity entering the hopper is reduced, causing the coarse particles to settle out. The gas enters the tube on the inside causing the fine particles to be collected, with the clean gas passing through the fabric into a common outlet manifold. After a certain amount of dust buildup, the flexible support mechanically shakes the particle loose from the fabric into the hopper. The reverse air cleaning baghouse operates in the same manner as the mechanical shaking arrangement, except a reverse air flow replaces the shaking process. An air vent located in the outlet manifold is opened allowing atmospheric air to enter the casing, thereby collapsing the bags and dislodging the dust particles. Baghouses incorporating the pulse jet cleaning method are constructed with an upper and lower compartment separated by a tube sheet. The upper portion serves as the discharge manifold. The felted filter bags are supported by a venturi-shaped thimble attached to the tube sheet. A compressed air jet is located above each filter bag to facilitate cleaning. Internal frames (mesh cages) with a closed bottom prevent the collapse of the bags during the cleaning cycle. Dirty gas enters the hopper and is then directed into the casing, passing through the filter bags. The dust is collected on the outside surface, allowing the clean air to pass through the fabric and out the discharge manifold. The filter bags are cleaned by the force of the pulse jet expanding the bags (see Figure 5).
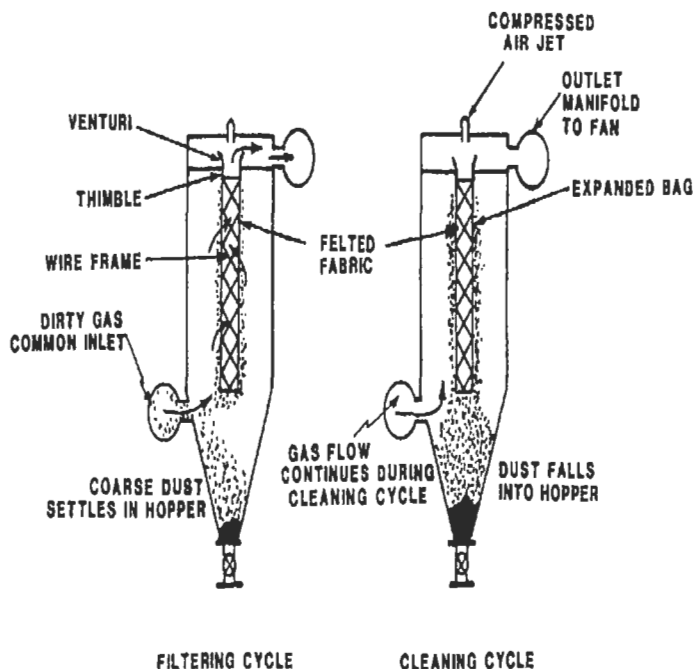
Figure 5. *Automatic baghouse with pulse jet cleaning.*

Filters are grouped according to the capacity by volume as follows: small volumes (i.e., below 10,000 acfm), medium volumes (i.e., 10,000 to 100,000 acfm), and large volumes (i.e., >100,000 acfm). The filter-media types include woven and felted media. Temperature capabilities of the media range from higher temperatures (> 400 °F), to medium temperatures (200 to 400 °F) and low temperatures (< 200 °F). The ability of the fabric to collect fine particles and maintain a good cleaning process should serve as the basis in selection of a fabric. As the dust layer or so-called filter cake layer builds up, flow resistance increases. Cleaning reduces the gas flow resistance and maintains the proper pressure drop across the filter.

Cloth filters are designed to remove three types of particles, and each type incorporates the basic principles of air filtration. Particles in the submicron size are collected as a result of the Brownian motion and bridging. As the particles build-up on the surface of the media, the collection surface areas increase causing particles to be captured. Collection efficiencies in excess of 99.95 % are possible. Particles having diameters in the 1 to 10 μ (micron) range and coarse particles (above 10 μ) rely on inertial collection. Efficiencies of 90 % to 95 % are achieved with particles under 10 μ in size. Efficiencies of coarse particles fluctuate from 50 % to 99.9 %. The reason for such variations in efficiency is a result of re-entrainment caused by gas flows at high velocities. High velocities can force the particles through the pores of the fabric.

Fabric filters are made of natural fibers, such as cotton and wool, or from synthetic fibers, depending upon their application. Cotton and wool are available in spun form, which limits the individual fibers to a few inches in length. Spun fibers can produce characteristics not found in filament (continuous) fibers. Filters composed of spun fibers are thicker, bulkier, heavier and provide a higher permeability to air flow. Synthetic fibers offer greater versatility such as higher operating ranges and corrosion resistance, but are more costly. Synthetic fibers are available as multi-filament fabrics, which are the most widely used and produced characteristics not common on spun forms. Multi-filament fabrics are light weight, of high tensile strength and high dimensional stability, abrasion resistant, and easy to clean.

Collection efficiency is affected by thread count, yarn size, and twist of the yarn. Permeability is increased by decreasing the thread count (either warp or fill), and subsequently increasing the pore area. Collection efficiency is reduced by increased permeability. A balanced weave is optimum for efficient operation. Permeability is also a function of the yarn size. The larger the yarn size, the lower the permeability. Yarn diameter, which can be altered by the twist of the yarn, also affects permeability; the smaller the diameter, the higher the permeability.

The ability of the gas stream to permeate the filter is also affected by the shrinking and elongation of the fabric. Elongation of the fibers results in increased pore space, and conversely shrinkage decreases the pore volume. Fabrics of good dimensional qualities are essential to fabric life and efficiency.

Fabric finishing steps enhance the collection efficiency of a system. The most commonly applied finishing steps include calendering, napping, singeing, glazing, and coating. Calendering is where surface fibers are pushed down onto the fabric medium by high pressure pressing on the fabric. Napping refers to scraping the filter medium, which raises the surface fibers. Singeing involves separate surface fibers that are removed by passing the filter medium over an open flame. Glazing involves surface fibers fused to the filter medium by high pressure pressing at elevated temperatures. Finally, coating involves a surface preparation over the fibers that reduces self-abrasion.

In order to specify a fabric filter, the properties of the incoming gas and particulate matter must be well defined. The properties of the gas essential to the problem definition are volume, temperature, moisture content, and acid gas concentration. The dust properties of importance are the density, particle size, particle size distribution, and the dust loading. The size of the fabric filter dust collector is directly related to the gas-to-cloth ratio as previously noted, which can be calculated by dividing the total gas volume by the total area of cloth or filter medium. Corrections to the gas-to-cloth ratio are required for the volume of air introduced to the unit and to account for the area of cloth not exposed to the incoming gas. The type of fabric selected is a function of the cleaning process employed. For example, the pulse-jet cleaning method would be used in conjunction with the felt fabric to achieve proper cleaning and efficiency. The filter medium type is the next major consideration following the gas/cloth ratio and cleaning method.

Cotton bags are used in standard installations and are the most economical. A maximum operating temperature of 180 °F is recommended for continuous use with 225 °F allowed for surge conditions. Wool bags are used for applications with dust particles of a combustible nature, or with operating temperatures of 200 °F and an allowable surge temperature of 250 °F. Nylon has a greater tensile strength than cotton or wool and provides excellent abrasion resistance. Fiberglass is most resistant to high temperatures, with a maximum operating temperature of 500 °F. To increase the allowable temperature, fiberglass filters are silicone treated to permit their use in applications such as in carbon black production plants. Replacement of bag filters generates the highest maintenance and cost of the system. Typical causes of bag failure include too high of a gas to cloth ratio, metal-to-cloth abrasion problems, chemical attack by the gas stream or particulates, inlet velocity abrasion, and excessive gas temperatures. The quality of the fabric and method of cleaning are additional factors to consider in evaluating service and maintenance costs. If a filter bag tears, it is important to repair the bag as quickly as possible to prevent abrasion to adjacent bags by jet streams of dust discharging out of the damaged bag. This type of bag failure is limited to inside bag collection types of dust collectors. The speed of repair is determined by the opacity of the outlet bag. In a compartmentalized system, broken bags can be found by monitoring the emissions while isolating one compartment at a time. To prevent a higher filter velocity, damaged filter bags within a compartment should not be replaced with clean bags. The higher velocity could create greater pressure drop or failure due to dust abrasion. An alternative is to plug or tie off the flow. To recap, the first step in selecting a fabric filter is to define the magnitude of the particulate loading. Knowledge of the particulate matter collected, properties of the gas stream, and the cleaning method are essential to proper design. Improper design leads to low efficiency and unscheduled maintenance. Prior to selection, results from a related application should be investigated. An alternative is to operate a pilot unit to ensure the most optimum gas-to-cloth ratio for a specified pressure drop. With proper design, operation, and maintenance, better than 99.9% efficiency can be achieved, depending on the application. A major advantage of fabric filters, is their ability to operate at a high efficiency at all loads from maximum down to very low gas flow. Some disadvantages of the system are the space requirements and high maintenance costs. Other problems associated with fabric filters are plugging of the fabric due to operation below the dew point or break down of the filter bags, resulting from high temperatures.

Fabric filters can be more costly to operate and maintain than electrostatic precipitators, cyclones, and scrubbers; however, fabric filters are more practicable for filtration of specific dusts. For example: fabric systems are the typical control method for toxic dusts from insecticide manufacturing processes, salt fumes from heat treating, metallic fumes from metallurgical processes, and other applications. Any other control method may not be as efficient, nor economically feasible for such applications.

Up to this point, we have described the operations and features of an industrial baghouse dust collector, which is essentially a system containing filter fabric of some kind which removes dust and particulate from a gas streams. The cleaned gas can then be vented to the atmosphere. If the dust is a by-product, the dust can be recycled back into the process. In contrast, a cartridge dust collector uses gravity together with a downward airflow pattern to provide extremely high filtration efficiency while reducing energy consumption. The cartridge is mounted in a horizontal and slightly downward sloping position. This design increases filter life and improves airflow because both gravity and airflow are pushing the dust downward into the collection hopper. Filters in baghouses typically need to be replaced after approximately one year of operation. Users should consider the cost of maintenance and filter replacement when purchasing or specifying a dust collector. This is an important cost factor to consider when comparing to cartridge filters which generally are less expensive. In addition, cartridge filters tend to be more efficient than a baghouse. A cartridge collector collects 99.9% of submicron particulate as compared to more typically 99.0% for a baghouse. This characteristic can be particularly important in applications involving toxic dust or when air is being recirculated.

On smaller filtration devices such as those used in fume control in indoor air applications, the real objective is to purify air through materials that trap contaminants which may include both particulate matter and gases. In such systems the fabric is impregnated with activated carbon or zeolite. Gases and odors filter through activated carbon and/or zeolite, an organic mineral. HEPA (high efficiency particulate air filter) is an effective medium for screening particles developed by the US Atomic Energy Commission. True medical rated HEPA is 99.97% effective in removing particles above 0.3 microns, or one three-hundredth the size of a human hair.

Clearly the largest application for filtration is air pollution control. On a worldwide basis the annual sales revenues for all types of pollution control equipment is around 150 billion dollars, with air pollution control devices being a substantial part of these equipment sales. The following is a summary of Web sites recommended to be consulted for specific vendor information on air filtration devices. A brief description of each site is provided. Many of these sites will link the reader to other sites containing additional information on product information.

Recommended Web Sites on Product Information for fabric filters and related devices:

- **Astec Microflow**: Manufacturers of fume cupboards, fume hoods and fume cabinets. Leaders in filtration and containment technology. *http://www.astec-microflow.co.uk*

- **TMS Air Filtration Systems Limited**: Specialist in air filtration, extraction and ventilation systems. *http://www.westmids.co.uk/tmsairfiltration*

and ventilation systems. *http://www.westmids.co.uk/tmsairfiltration*

- **Munktell Filter AB**: Highly qualified producer of filter media in medical, industrial, environmental controlling and analytical, and purifying filtration of air, fumes and fluids. *http://www.munktell.se*

- **Coppys**: Provider of portable ventilators which help fume and vapor removal, compressed air filtration, personnel cooling & confined space ventilation with propane heaters, cooling mist & ventilation blowers. http://www.coppus.com

- **Airflow Systems, Inc**: Resource for the collection and filtration of dust, smoke, mist, fumes and other airborne contaminants generated during industrial and commercial manufacturing and processing applications. *http.,llwww.air,flowsystems.com*

- **Clean Air Ltd**: Fume cabinets (or cupboards, UK) designed and produced quickly and efficiently for a wide range of Research, Pharmaceuticals, Chemicals, or educational settings. *http://www.cleanairltd.co.uk*

- **Campbell Environmental Systems**: Sells Austin air HEPA air filters and Miele HEPA vacuum cleaners *http://www.airwaterbestprices-comlindex.html*

- **Air Cleaning Systems, Inc.**: Sells, services and provides equipment and parts for air filtration systems. *http://www.aircleaningsystems.com*

- **Conquest Equipment Corporation**: Industrial filtration products and services to clients in the wood, paper, plastics, metal, aerospace, and mining industries. *http://www.conquestequipment.com*

- **Hoffman & Hoffman, Inc.**: Manufacturers sales representatives in the selection and application of commercial heating, ventilation, air conditioning, filtration and DDC systems. *http://www.hoffman-hoffman.com*

- **Clean Air Machine Corp.**: Air purifiers - commercial site offers customized filters for specialty applications aimed at removal of microscopic particulates and gases. *http://aco.ca/tibbits/index .html*

- **Aircon Corp.**: Site markets air pollution systems including dust control systems and equipment. *http://aircon-corporation.com*

- CECO Environmental: Experts in improving air quality through the use of fiber bed filter systems, high temperature baghouse filter fabrics, scrubbing technology, and on-site air quality monitoring. *http://www.cecofilters.com*

- **Fortress Designs**: Supplier of dust control equipment, baghouses, dust collectors, fabric filters, dust filters, bin vents. *http://www.fabricfilters.com*

- **Schrader Environmental Systems, Inc.**: Air pollution control technologies such as catalytic oxidizers, packed tower wet scrubbers, dust collectors. *http://www.angelfire.com*

orientation, Table 1 provides an equipment selection guide that can be used in selecting the most appropriate piece of equipment for a gas cleaning application. The table is based upon general vendor literature reported in terms of removal efficiency and general applicability to a gas cleaning problem.

Table 1. *Equipment Selection Guide.*

Particle Size	Small	Tiny	Invisible	Submicron
Particle Type	> 1/64", 20 mesh	4/1000", 140 mesh	4/100,000" - 8/10,000"	< 4/100,000"
Size =	850 microns	51 - 850 microns	2 - 50 microns	< 1 micron
Solids Particulates	Gravity settler	Gravity settler, Cylcone	Coarse (Grit) filter, Cyclone	Cartridge filter
Liquid Particulates	Cyclone	Gravity settler	Cyclone, Baghouse	Scrubber
Combination	Fume filters, Scrubber	Fume filters, Scrubber	Mist and fume filters	Scrubber

Liquid Filtration Equipment

Liquid filtration equipment is commercially available in a wide range. Proper selection must be based on detailed information of the slurry to be handled, cake properties, anticipated capacities and process operating conditions. One may then select the preferred operational mode (batch, sem-batch or continuous), and choose a particular system on the above considerations and economic constraints. Continuous filters are comprised of essentially a large number of elemental surfaces, on which different operations are performed. These operations performed in series are solids separation and cake formation, cake washing, cake dewatering and drying, cake removal, and filter media washing. The specific equipment used can be classified into two groups: (1) stationary components (which are the supporting devices such as the suspension vessel); and (2) scraping mechanisms and movable devices (which can be the filter medium, depending on the design). Either continuous or batch filters can be employed in cake filtration. In filter-medium filtration, however, where particulate matter are retained within the framework of the filter medium, batch systems are the most common. Batch filters may be operated in any filtration regime, whereas continuous filters are most often operated under constant pressure. It is important to note that equipment used in liquid filtration applications fall into three general operational modes based on the

difference in orientation between gravity force and filtrate motion. These orientations are: forces acting in the opposite direction (i.e, countercurrent systems), forces acting in the same direction (cocurrent), and forces acting normal to each other (cross-flow).

Solid-liquid separation is a fundamental unit operation that exists in almost every flowscheme related to the chemical process industries, ore beneficiation, pharmaceutics, food or drinking water and wastewater treatment. The separation techniques are very diverse. As a unit operation, there are many types of equipment that are used, not just filtration systems. In addition to filtration by such equipment as vacuum and pressure filters described below, there are centrifugation by filtering and sedimenting centrifuges, sedimentation by conventional, storage and high-rate thickeners, clarification by conventional, solids-contact and sludge-blanket clarifiers, polishing by precoat filters, pressure and deep-bed filters, and upward separation by dissolved-air flotation .

The first group of liquid filtration equipment described is the vacuum filter. Vacuum filters are relatively simple and reliable machines, and as such they have gained wide acceptance in the chemical processing and allied industries. The various types of vacuum filters are summarized in the chart provided in Figure 6. For a sense of physical dimensions, the following are some typical sizes: drum and disc filters are up to 100 m^2 in filtration area; horizontal belt filters up to 120 m^2; tilting pan and table filters up to 200 m^2. A typical filter station is comprised of the following components and the flowsheet can look like the example provided in Figure 7:

- The vacuum filter

- One or more vacuum receivers (if separation of the mother filtrate from the wash filtrate is needed)

- One or more filter pumps (either receiver mounted or connected to the seal tank of a barometric leg)

- Vacuum pump (either liquid ring or rotating vanes type)

- Piping and instrumentation

There are several advantages that vacuum filtration has over other solid-liquid separation methods. Some of these advantages include:

- Continuous operation (with the exception of the Nutsche type filter),

- Intensive soluble recovery or removal of contaminants from the cake as accomplished by countercurrent washing operations. This is especially the case with horizontal belt, tilting pan and table filters, which are described later in this subsection,

- Polishing of solutions (as performed on a precoat filter operation),
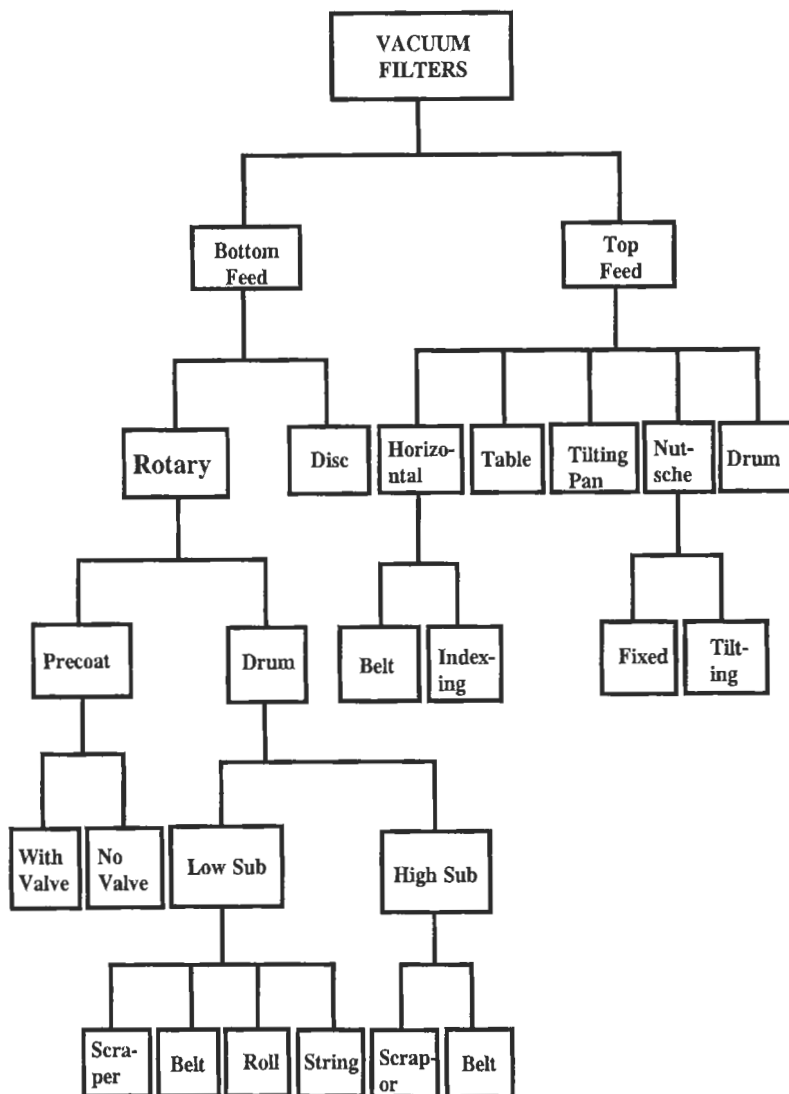
Figure 6. *Organizational chart for vacuum filters.*

Additional advantages that vacuum filters have over other separation methods is the ability ti produce relatively clean filtrates by using a cloudy port or a sedimentation basin, as in the cases with the horizontal belt, tilting pan and table filters; they provide easy access to cake for sampling purposes; they allow easy control of operating variables such as cake thickness and wash rations; commercial systems are available in a wide range of materials of construction. The principle disadvantages of vacuum filters include higher residual moisture in the cake, untight

construction so it is difficult to contain process vapors, and high power requirements as demanded by the vacuum pump.

One of the oldest filters applied throughout the chemical processing industry is the rotary vacuum drum filter, which is illustrated in Figure 8. This machine belongs to the group of bottom feed configurations. Rotary drum filters are typically operated in the countercurrent mode of operation. The principle advantage of these machines is the continuity of their operation.
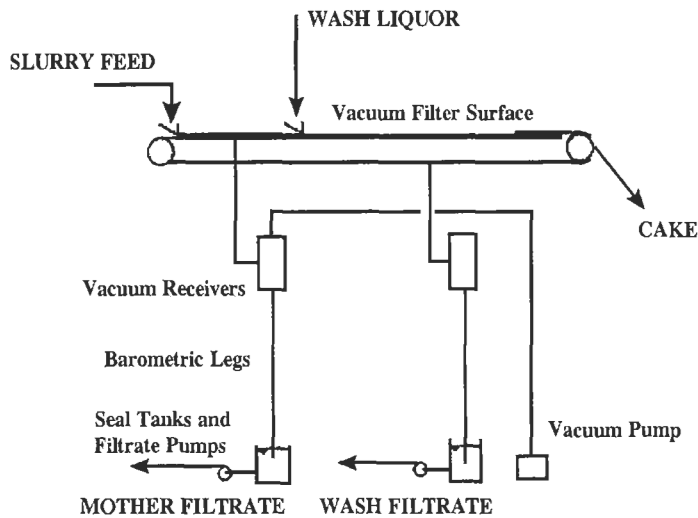
Figure 7. *Example of filtration flow scheme.*
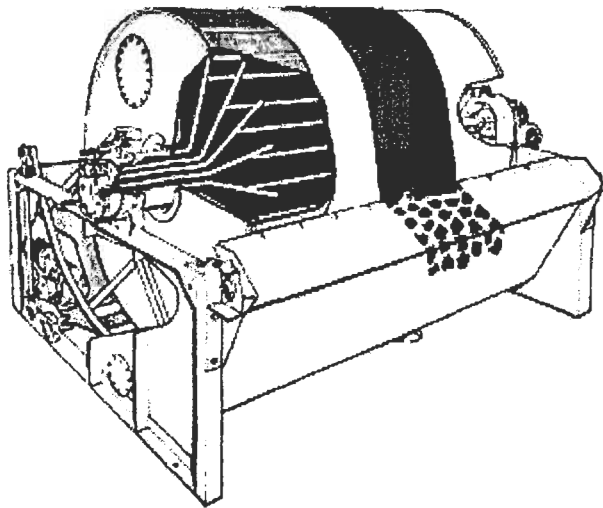
Figure 8. Rotary drum filter.

The key features of the a rotary drum filter are as follows. First, the machine has a drum that is supported by a large diameter trunnion on the valve end and a bearing on the drive end, and its face is divided into circumferential sectors each forming a separate vacuum cell. The internal piping that is connected to each sector passes through the trunnion and ends up with a wear plate having openings which correspond to the number of sectors. The drum deck piping is arranged so that each sector has a leading pipe to collect the filtrate on the rising side of the drum and a trailing pipe to collect the remaining filtrate from the descending side to ensure complete evacuation prior to cake discharge. The drum is driven with a variable speed drive at speeds that normally range from 1 to 10 MPR. The machine is equipped with a valve with a bridge setting which controls the sequence of the cycle so that each sector is subjected to vacuum, blow and a dead zone. When a sector enters submergence, vacuum commences and continues to a point that it is cut-off and blow takes place to assist in discharging the cake. The valve has on certain filters adjustable blocks and on others a fixed bridge ring. Adjustable bridge blocks enable the optimization of form to dry ratio within the filtration cycle as well as the submergence of the drum in the tank. A cake discharge mechanism is available in several forms, such as a scraper, belt, roll and in very rare cases a string discharge. Blow is applied only to filters with scraper and roll discharge mechanisms but not to filters with a belt or string discharge. The various types of cake discharge mechanisms are illustrated in Figures 9 through 12.
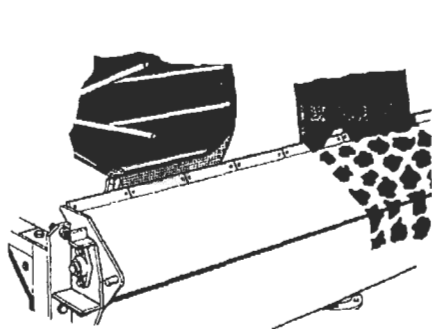
Figure 9. *Shows scraper discharge.*
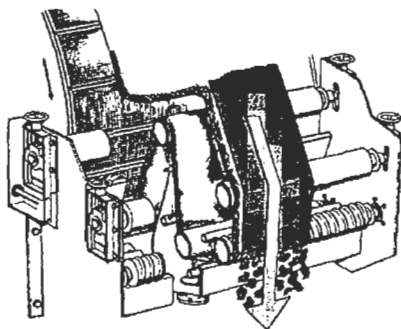
Figure 10. *Shows a belt discharge.*
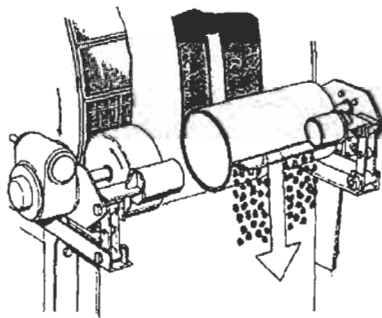
Figure 11. *Shows a roll discharge.*
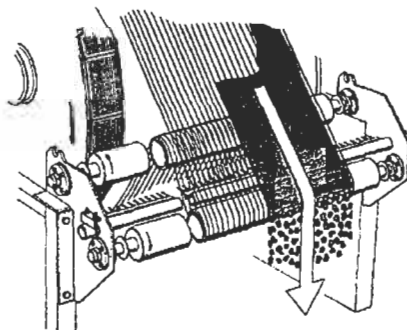
Figure 12.. *Shows a string discharge.*

A mild agitator is used to keep the slurry in suspension and reciprocates between the drum face and tank bottom at 16 or so CPM. The system is equipped with a tank with baffled slurry feed connections, an adjustable overflow box to set a desired drum submergence and a drain connection. The tanks are normally designed for an apparent submergence of 33 to 35%, however on certain applications 50% and more is possible. With these special designs the tank ends are higher in order to accommodate stuffing boxes on both the drive shaft and valve end trunnion. On applications where cake washing is required, 2 or 3 manifolds with overlapping nozzles are mounted to a pair of splash guards that are bolted to the tank ends. The position of the manifolds and the quantity of wash liquid are adjustable depending on the wash characteristics of the cake. Optional controls may be used to automate settings such as drum speed, applied wash liquid and drum submergence for a desired cake thickness or throughput. The monitoring of drum submergence controls the slurry feed valves so an adjustable overflow weir is not necessary except for a fixed connection in case of emergency. In broad terms, drum filters are suitable to the following process requirements:

- Slurries with solids that do not tend to settle rapidly and will remain in a uniform suspension under gentle agitation,

- Cakes which do not require long drying times to reach asymptotic moisture values,

- Cakes when a single washing stage is sufficient to remove residual contaminants from the cake or yield maximum recovery of filtrate,

- Filtrates that generally do not require a sharp separation between the mother and wash filtrates. Some complex valves, however, enable atmospheric purging of the sectors and internal piping to facilitate a sharp separation of filtrates,

- Filtrates that are acceptable with a low quantity of fines that pass trough the filter cloth in the first few seconds of cake formation. Broadly, and depending on particle size and cloth permeability, the filtrate may contain 1,000 to 5,000 ppm insolubles.

The entire filtration cycle on a rotary drum filter must be completed within a geometry of 360 degrees. The cycle sequence of a single sector is illustrated in Figure 13, where it is assumed that the drum rotates in a clockwise direction. The stages of the cycles are cake formation, cake washing and drying, cake discharge, and the dead zone. Each is briefly described below.

Cake Formation: With the overflow weir set to a maximum the "apparent submergence" is normally 33 to 35% so the slurry levels between 0400 and 0800 hours. Once a sector is completely submerged vacuum is applied and a cake starts to form up to a point where the sector emerges from the slurry. The portion of the cycle available for formation is the "effective submergence" and its duration

depends on the number of sectors, the slurry level in the tank and the bridge setting which controls the form to dry ratio.

Cake Washing and Drying: After emerging from submergence the drying portion of the cycle commences and for non-wash applications continuous to about 0130 hours where the vacuum is cut-off. If cake washing is required the wash manifolds will be located from about 1030 to 1130 hrs and the remaining time to vacuum cut-off at 0130 is the portion allocated to final cake drying.

Cake Discharge: After vacuum for the entire sector is cut-off air blow commences at about 0200 hours in order to facilitate the discharge of the cake. The blow, depending on the position of the tip of the scraper blade, will cut-off at approximately 0300 hrs. Drum filters are normally operated with a low pressure blow but on certain applications a snap blow is applied and to avoid the snapping out of the caulking bars or ropes wire winding of the cloth is recommended . Blow is used on scraper and roll discharge mechanisms but on belt discharge filters vacuum cuts-off when the filter media leaves the drum.

Dead Zone: Once the blow is cut-off the sector passes through a zone blocked with bridges so that no air is drawn through the exposed filter media which might cause the loss of vacuum on the entire drum surface.

The slow rotation of the drum and reciprocation of the agitator reduce maintenance requirements to a minimum but the following should be inspected periodically. The strip liner of the trunnion bearing at the valve end will normally wear at the lower half. However, in situations when the slurry has a high specific gravity, the drum has a tendency to become buoyant causing wear to the upper half.
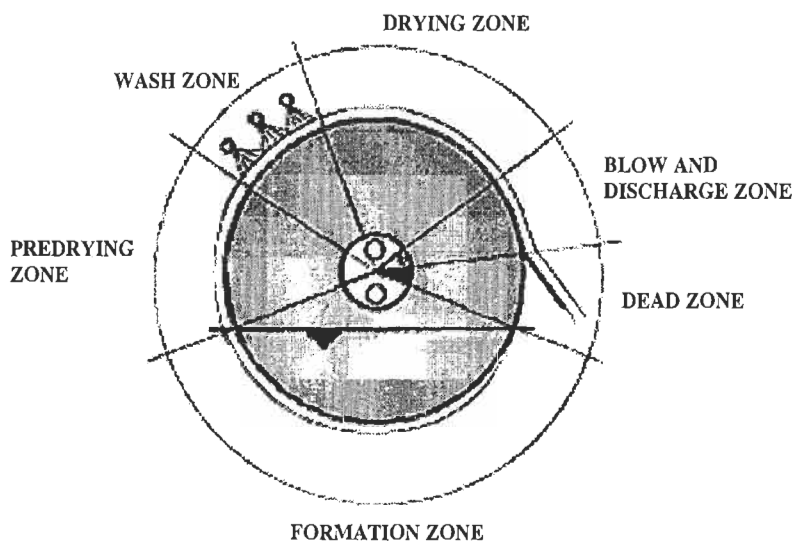
Figure 13. *Cycle sequence of a rotary drum filter.*

One way to remove the lower half of the liner, when hoisting facilities are not available or operational, is to float the drum by filling the tank with a sufficiently concentrated solution. Other components that should be periodically checked are:

- The stuffing boxes on high submergence filters should be inspected for leakage and, if necessary, the stud nuts should be tightened. It should be noted that excess tightening can increase substantially the load on the drum drive so the use of a torque wrench is recommended.

- The face of the wear plate should be checked periodically and remachined if necessary. A whistling noise during operation is an indication the wear plate is worn out or the valve spring requires tensioning.

- The drum has a bailer tube that protrudes from the drive end shaft and must be kept open at all times since its blockage may cause the collapse of the drum. The bailer tube is a tell-tale indication to the following: (a) if a lighter flame is drawn through the bailer tube to the inside of the drum it indicates that a vacuum leak exists in the drum shell or the internal piping; (b) if leakage is observed from the bailer tube it indicates that a hole exists in the drum head causing penetration of slurry from the tank into the drum.

- The on-line filter on the wash headers manifold should be checked periodically for pressure build- up due to progressive blockage. Likewise, the nozzles on the wash headers should be kept clean in order to ensure overlapping for full coverage of the washed cake.

Internal Rotary-Drum Filters: An example of an internal rotary-drum filter is illustrated in Figure 14. The filter medium is contained on the inner periphery. This design is ideal for rapidly settling slurries that do not require a high degree of washing. Tankless filters of this design consist of multiple-compartment drum vacuum filters.
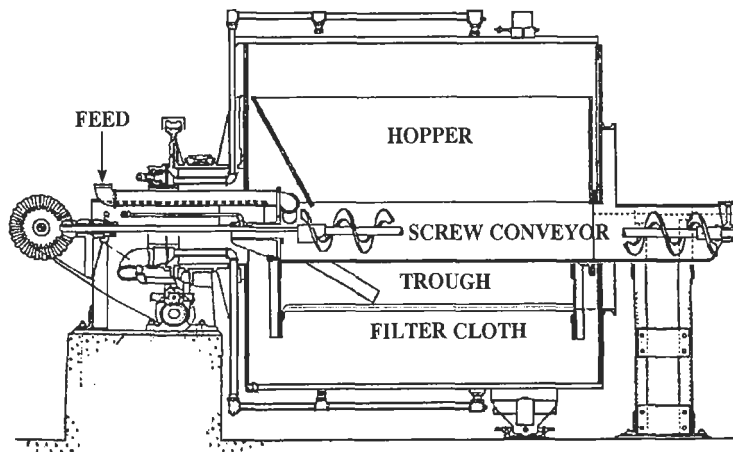
Figure 14. *Section view of interior medium rotary-drum vacuum filter.*

One end is closed and contains an automatic valve with pipe connections to individual compartments. The other end is open for the feed entrance. The drum is supported on a tire with rigid rollers to effect cake removal. The drum is driven by a motor and speed-reducer connected to a riding roll shaft. The feed slurry is discharged to the bottom of the inside of the drum from the distributor and is maintained as a pool by a baffle ring located around the open end and the closed portion of the outer end. As the drum revolves, the compartments successively pass through the slurry pool, where a vacuum is applied as each compartment becomes submerged. Slurry discharge is accomplished at the top center where the vacuum is cut off and gravity (usually assisted by blowback) allows the solids to drop off onto a trough. From there, a screw or belt conveyor removes the solids from the drum. This filter is capable of handling heavy, quick-settling materials.

Nutsche Filters: Nutsche filters are one design type with a flat filtering plate. This configuration basically consists of a large false-bottomed tank with a loose filter medium. Older designs employ sand or other loose, inert materials as the filtering medium, and are widely employed in water clarification operations. In vacuum filtration, these false-bottom tanks are of the same general design as the vessels employed for gravity filtration. They are, however, less widely used, being confined for the most part to rather small units, particularly for acid work. Greater strength and more careful construction are necessary to withstand the higher pressure differentials of vacuum over gravity. This naturally increases construction costs. However, when high filtering capacity or rapid handling is required with the use of vacuum, the advantages may more than offset higher costs. Construction of the vacuum false-bottom tank is relatively simple; a single vessel is divided into two chambers by a perforated section. The upper chamber operates under atmospheric pressure and retains the unfiltered slurry. The perforated false bottom supports the filter medium. The lower chamber is designed for negative pressure, and to hold the filtrate. Nutsche filters are capable of providing frequent and uniform washings. A type of continuous filter that essentially consists of a series of Nutsche filters is the rotating-tray horizontal filter.

Horizontal Rotary Filters: An example of a horizontal rotary filter is illustrated in Figure 15. These machines are well suited to filtering quick-draining crystalline-like solids. Due to its horizontal surface, solids are prevented from falling off or from being washed off by the wash water. As such, an unusually heavy layer of solids can be tolerated. The basic design consists of a circular horizontal table that rotates about a center axis. The table is comprised of a number of hollow pie-shaped segments with perforated or woven metal tops. Each of the sections is covered with a suitable filter medium and is connected to a central valve mechanism that appropriately times the removal of filtrate and wash liquids and the dewatering of the cake during each revolution. Each segment receives the slurry in succession. The wash liquor is sprayed onto each section in two applications. Then the cake is dewatered by passing dry air over it. The cake is finally removed by a scraper.
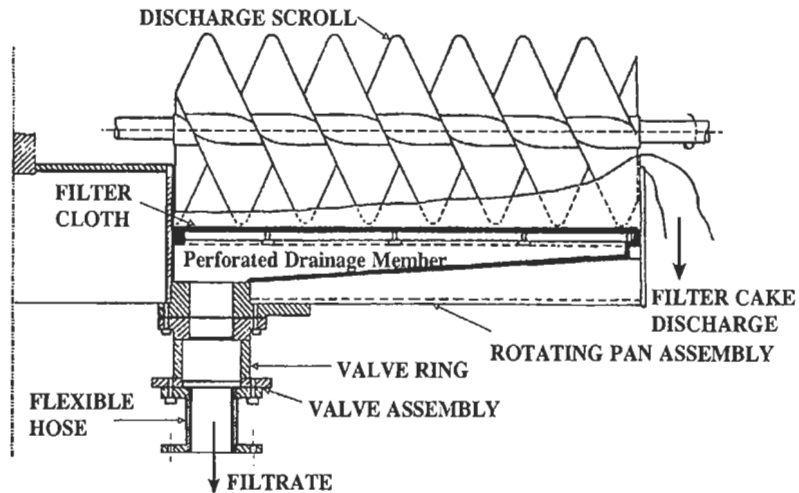
Figure 15. *Cross section of a rotary horizontal vacuum filter.*

Belt Filters: Belt filters consist of a series of Nutsche filters moving along a closed path. Nutsche filters are connected as a long chain so that the longitudinal edge of each unit has the shape of a baffle plate overlapping the edge of the neighboring unit. Each unit is displaced by driving and tensioning drums. Nutsche filters are equipped with supporting perforated partitions covered with the filtering cloth. The washed cake is removed by turning each unit over. Sometimes a shaker mechanism is included to ensure more complete cake removal. A belt filter can be thought of as consisting of an endless supporting perforated rubber belt covered with the filtering cloth. The basic design is illustrated in Figure 16. Supporting and filtering partitions are displaced by driving drum and maintained in a stretched condition by tensioning the drum, which rotates due to friction against the rubber belt. Belt edges (at the upper part of their path) slide over two parallel horizontal guide planks. The elongated chamber is located between the guide planks. The chamber in the upper part has grids with flanges adjoining the lower surface of the rubber belt. The region under the belt is connected by nozzles to the filtrate collector, which is attached to a vacuum source. The chamber and collector are divided into sections from which filtrate and washing liquid may be discharged. The sludge is fed by the trough. The cake is removed from the drum by gravity or blowing air, or sometimes it is washed off by liquid from the distributor nozzle. The washing liquid is supplied from a dike tank, which can move along the filtering partition. It can be washed during the belt's motion along the lower path. The filtering partition, illustrated in Figure 17, consists of a riffled rubber belt with slots, grooves and the filter cloth, which is fixed in a set of grooves by cords. Slots through which the filtrate passes are located over the grids of the elongated chamber. The edges of the rubber belt are bent upward by guides forming a gutter on the upper path of the

belt. The velocity of the filtering partition depends on the physical properties of the sludge and the filter length. The cake thickness may range from 1 to 25 turns. The advantages of belt filters are their simplicity in design compared to filters with automatic valves, and the abilities to provide countercurrent cake washing and removal of thin layers of cake. Their disadvantages include large area requirements, inefficient use of the total available filter area, and poor washing at the belt edges.

Cross Mode Filters: Filters of this group have a vertical flat or cylindrical filtering partition. In this case, filtrate may move inside the channels of the filtering elements along the surface of the filtering partition downward under gravity force action, or rise along this partition upward under the action of a pressure differential. In the separation of heterogeneous suspensions, nonuniform cake formation along the height can occur because larger particles tend to settle out first. This often results in poor cake washing due to different specific resistance over the partition height.
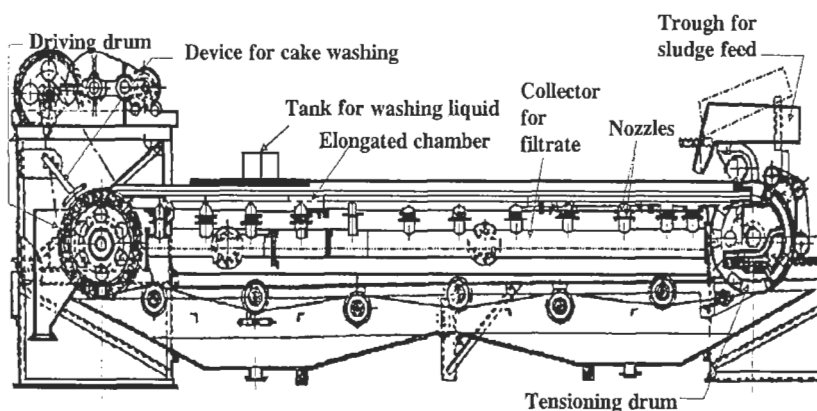
Figure 16. *Basic design features of a belt filter.*
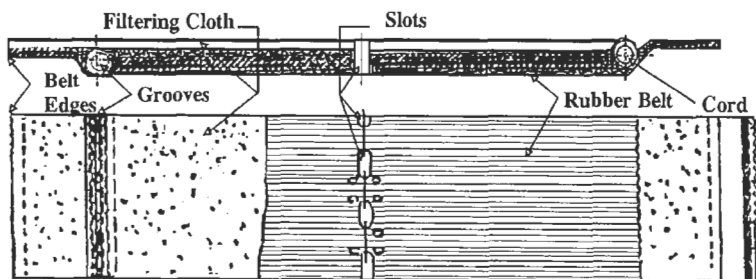
Figure 17. *Filtering partition for a belt filter.*

The cake may creep down along the partition due to gravity; this is almost inevitable in the absence of a pressure gradient across the filtering partition. The vertical filtering partition makes these filters especially useful as thickeners, since it is convenient to remove cake by reverse filtrate flow.

Filter Presses: The most common filter press is the plate-and-frame design, consisting of a metal frame made up of two end supports rigidly held together by two horizontal steel bars. Varying numbers of flat plates containing cloth filter media are positioned on these bars. The number of plates depends on the desired capacity and cake thickness. The plates are clamped together so that their frames are flush against each other, forming a series of hollow chambers. The faces of the plates are grooved, either pyramided or ribbed. The entire plate is covered with cloth, which forms the filtering surface. The filter cloth has holes that register with the connections on the plates and frames, so that when the press is assembled these openings form a continuous channel over the entire length of the press and register with the corresponding connections on the fixed head. The channel opens only into the interior of the frames and has no openings on the plates. At the bottom of the plates, holes are cored so that they connect the faces of the plates to the outlet cocks. As the filterable slurry is pumped through the feed channel, it first fills all of the frames. As the feed pump continues to supply fluid and build up pressure, the filtrate passes through the cloth, runs down the face of the plate and passes out through the discharge cock. When the press is full, it is opened and dumped. The cake cannot be washed in these units and is therefore discharged containing a certain amount of filtrate with whatever valuable or undesirable material it may contain. Each plate discharges a visible stream of filtrate into the collecting launder. Hence, if any cloth breaks or runs cloudy, that plate can be shut off without spoiling the entire batch. If the solids are to be recovered, the cake is usually washed. In this case, the filter has a separate wash feed line. The plates consist of washing and nonwashing types arranged alternately, starting with the head plate as the first nonwashing plate. The wash liquor moves down the channels along the side of each washing plate, and moves across the filter cake to the opposite plate and drains toward the outlet. This is illustrated in Figure 18. To simplify assembly, the nonwashing plates are marked with one button and the washing plates with three buttons. The frames carry two buttons. In open-delivery filters the cocks on the one-button plates remain open and those on the three-button plates are closed. In closed-delivery filters a separate wash outlet conduit is provided. Figure 19 illustrates the basic design of a frame, a nonwashing plate and a washing plate. Initial capital investment and floor space requirements are low in comparison to other types of filters. They can be operated at fall capacity (i.e., all frames in use) or at reduced capacity by blanking off some of the frames by dummy or blank plates. They can deliver reasonably well washed and relatively dry cakes. However, the combination of labor charges for removing the cakes and fixed charges for downtime may constitute a high percentage of the total cost per operating cycle.
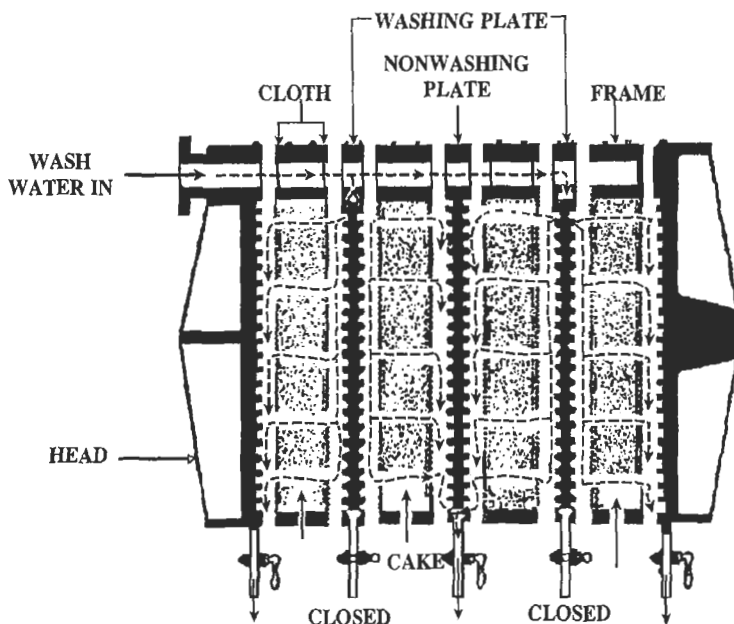
Figure 18. *Wash water outlets on a filter press.*
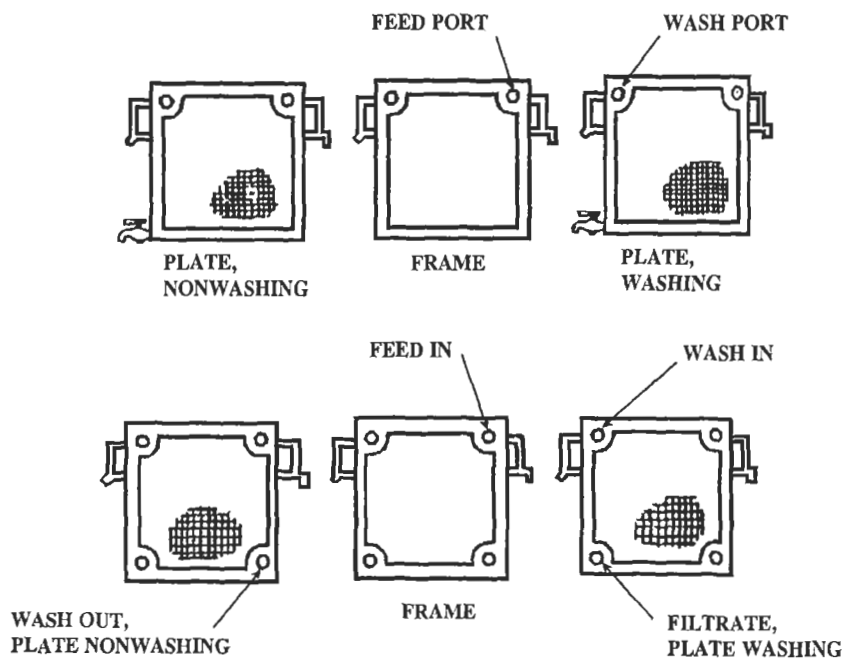
Figure 19. *Plate and frames for open and closed delivery systems.*

Leaf Filters: Leaf filters are similar to plate-and-frame filters in that a cake is deposited on each side of a leaf and the filtrate flows to the outlet in the channels provided by a coarse drainage screen in the leaf between the cakes. Figure 20 illustrates the basic scheme. The leaves are immersed in the sludge when filtering, and in the wash liquid when washing. Therefore, the leaf assembly may be enclosed in a shell, as in pressure filtration, or simply immersed in sludge contained in an open tank, as in vacuum, filtration. When operating a pressure leaf filter, the sludge is fed under pressure from the bottom and is equally distributed. The clear filtrate from each leaf is collected in a common manifold and carried away. In filters with an external filtrate manifold as illustrated in Figure 21, the filtrate from each leaf is visible through a respective sight glass. This is not possible when the leaves are mounted on a hollow shaft that serves as an internal filtrate collecting manifold. The filter cakes build on each side of the leaves as filtration continues, until a specified cake thickness is achieved. During the cake washing phase, excess sludge is first drained while compressed air is introduced at about 3 to 5 psi. The role of the compressed air is to prevent the cake from pealing off of the leaves during the washing stage.
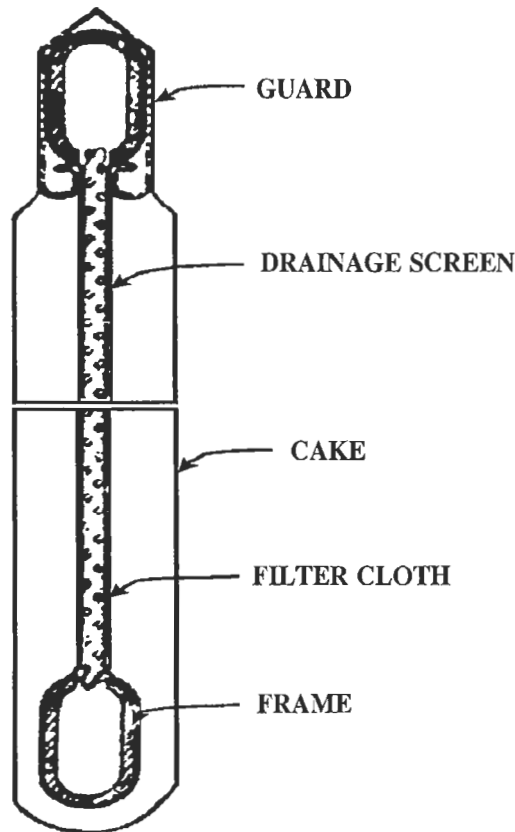
Figure 20. *Sectional view.*
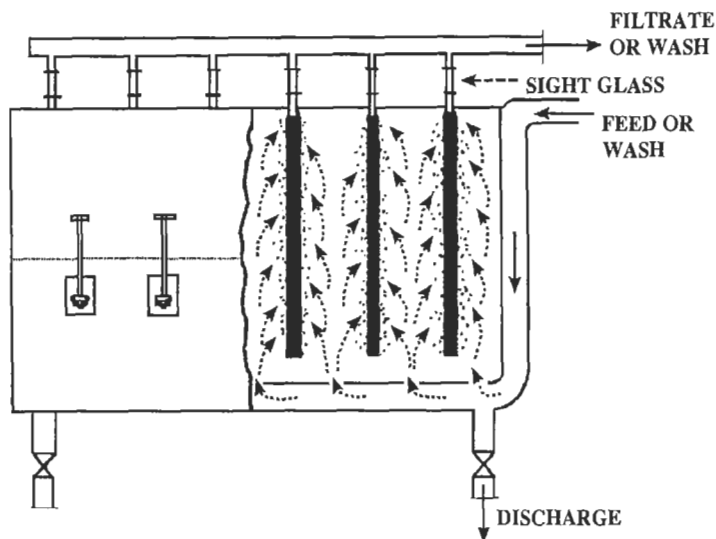
Figure 21. *Sweetland pressure filter.*
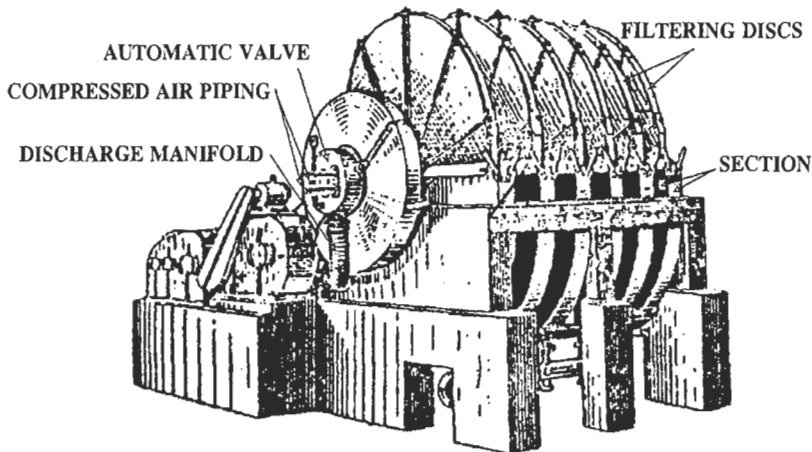
Figure 22. *Rotary-disc vacuum filter.*

Disk Filters: Disk filters consist of a number of concentric disks mounted on a horizontal rotary shaft. The operating principle is the same as that of rotary-drum vacuum filters. The basic design is illustrated in Figure 22. The disks are formed by using V-shaped hollow sectors assembled radially about a central shaft. Each sector is covered with filter cloth and has an outlet nipple connected to a manifold

extending along the length of the shaft and leading to a port on the filter valve. Each row of sectors is connected to a separate manifold. The sludge level in the tank should provide complete submergence to the lowest sector of the disks. Compared to drum vacuum filters, disk filters require considerably less floor space for the same filter area. However, because these machines have vertical filtering surfaces, cake washing tends not to be as efficient as in the case of a drum filter. The disk filter is best suited for cases when the cake does not require washing and floor space is critical.

Cartridge Filters: Cartridge filters are used in a multitude of solid-liquid filtration applications ranging from laboratory scale operations to industrial flows in excess of 5,000 gpm. These units are typically operated in the countercurrent mode. Common configurations consist of a series of thin metal disks that are 3 to 10 inches in diameter, set in a vertical stack with very narrow uniform spaces between them. The disks are supported on a vertical hollow shaft, and fit into a closed cylindrical casing. Liquid is fed to the casing under pressure, whence it flows inward between the disks to openings in the central shaft and out through the top of the casing. Solid particles are captured between the disks and remain on the filter media. Since most of the solids are removed at the periphery of the disks, the unit is referred to as an edge filter. The accumulated solids are periodically removed from the cartridge. As with any filter, careful media selection is critical. Media that are too coarse, for example, will not provide the needed protection. However, specifying finer media than necessary can add substantially to both equipment and operating costs. Factors to be considered in media selection include the solids loading, the nature and properties of the particles, particle size, shape and size distribution, the amount of solids to be filtered, fluid viscosity, slurry corrosiveness, abrasiveness, adhesive qualities, liquid temperature, and flowrate. Typical filter media are wire mesh (typically 10 to 700 mesh), fabric (30 mesh - 1 μ), slotted screens (10 mesh to 25 μ) and perforated stainless steel screens (10 to 30 mesh). Multiple filters are also common, consisting of two or more single filter units valved in parallel to common headers. The distinguishing feature of these filters is the ability to sequentially backwash each unit in place while the others remain on stream. Hence, these systems are continuous filters. These units can be fully automated to eliminate manual backwashing. Backwashing can be controlled by changes in differential pressure between the inlet and outlet headers. One possible arrangement consists of a controller and solenoid valves that supply air signals to pneumatic valve actuators on each individual filter unit. As solids collect on the filter elements, flow resistance increases. This increases the pressure differential across the elements and, thus, between the inlet and outlet headers on the system. When the pressure drop reaches a preset level, an adjustable differential pressure switch relays information through a programmer to a set of solenoid valves, which in turn sends a signal to the valve actuator. This rotates the necessary valve(s) to backwash the first filter element. When the first element is cleaned and back on stream, each successive filter element

is backwashed in sequence until they are all cleaned. The programmer is then automatically reset until the rising differential pressure again initiates the backwashing cycle. Filter cartridges or tubes are made from a variety of materials. Common designs are natural or synthetic fiber wound over a perforated plastic or metal core. A precision winding pattern covers the entire depth of the filter tube with hundreds of funnel-shaped tunnels, which become gradually finer from the outer surface to the center of the tube and trap progressively finer particles as the fluid travels to the center. This provides greater solids retention capacity than is associated with surface filter media of the same dimensions. Typical cartridge materials are cotton, Dynel, polypropylene, acetate, porous stone and porous carbon filter lubes. Supporting perforated cores for cotton, Dynel or polypropylene are stainless steel, polypropylene or steel. Supporting cores for acetate tubes are tin-plated copper with voile liner. Porous stone and porous carbon filter tubes do not require supporting cores. Stainless steel cores are recommended for mildly acid and all alkaline solutions, i.e., pH 4-14. Polypropylene cores are used where all metal contact must be eliminated or where stainless steel is attacked, such as high chloride and sulfuric acid solutions. Two types of polypropylene cores are commonly used: mesh polypropylene and rigid perforated polypropylene. Mesh polypropylene is satisfactory for temperatures below 140 °F. The more expensive rigid polypropylene cores are used for temperature applications over 140 °F, and for double- and triple-tiered filter chambers because greater strength is needed here. Perforated steel cores are used for dilute alkaline solutions, solvents, lacquers, oils, emulsions, etc.

Strainers and Filter Baskets: Strainer filter baskets and filter bag baskets are used as prefiltering devices. This prestraining or prefiltering prevents larger contaminated particles from continuing through the filtration process and thus extends the life of the entire system. Single-stage strainers and bag filters differ only in the basket design. Strainer baskets have solid flat bottoms, and baskets for filter bags have perforated bottoms to accept standard size filter bags. Dual-stage straining/ filtering action is achieved by insertion of a second, inner basket. It is supported on the top flange of the outer basket. Both baskets can be strainers (with or without wire mesh linings) or both can be baskets for filter bags. They may also be a combination: one a strainer basket, the other a filter bag basket. Dual-stage action increases strainer or filter life and reduces servicing needs. All baskets are equipped with a seal. The seal is maintained during operation by a hinged basket bail handle being held down under the closed cover, which holds the basket down against a positive stop in the housing. Figure 23 illustrates the design features. Fabric bag filter baskets are capable of providing removal ratings from 20 mesh to nominal 1 micron (μ) for both Newtonian and viscous liquids. Wire mesh or fabric baskets can be cleaned and reused in many applications, or are disposable when cleaning is not feasible. Side-entry models feature permanent flanged connections, for line pressures to 150 psi. These filters are fabricated to American Society of Mechanical Engineers (ASME) codes for applications that must comply with piping

standards established in many processing plants. Top-entry models feature the inlet connection as an integral part of the lid. The inlet can be equipped with different types of quick disconnects for fast basket removal. Strainers should be selected so that the pressure drop incurred does not exceed a specified limit with a clean strainer basket (typically 2 psi). Pressure drop versus flow capacity curves for basket strainers are given in Figure 24. This plot provides gross pressure drop for different capacities of water flow at suitable strainer pipe sizes. The value obtained must be corrected on the basis of the actual fluid viscosity and strainer opening size to be used. These corrections are given in table accompanying the plot in Figure 24, using the following stepwise procedure - Step (1): Under the pressure drop value from the bottom scale in Figure 24, with the specified flowrate - read up to where its vertical line intersects the diagonal representing a strainer pipe size that gives a reasonable pressure drop, which is found by following the horizontal line to the pressure drop scale at the left. Step (2): To correct this figure to match the actual fluid viscosity (and strainer media selection), use the table values with Figure 24. Read down the appropriate viscosity column and across from the appropriate strainer media description to obtain the correction factor. Step (3): Multiply the pressure drop figure obtained under step 2 by the correction factor to obtain the adjusted pressure drop. Step (4): If you are using a mesh-lined (not pleated) strainer basket, the pressure drop can be lowered by using a 30-in.-deep basket instead of a 15-in.-deep basket; divide the pressure drop figure obtained from step 3 by 1.5.

Diaphragm Filters: Diaphragm filters are special case of filter presses. That have the ability to reduce sludge dewatering costs by a squeezing cycle using a diaphragm. Instead of the conventional plate-and-frame unit in which constant pumping pressure is used to force the filtrate through the cloth, diaphragm filters combine an initial pumping followed by a squeezing cycle that can reduce the process cycle time by as much as 80%. The operating cycles for this design are illustrated in Figure 25.
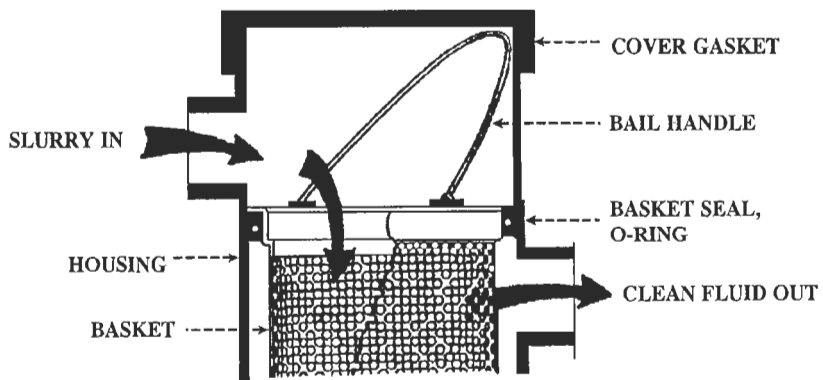
Figure 23. *Upper portion of basket with seal.*

STRAINER PIPE SIZE   2"   3"   4"

FLOWRATE, GPM

VISCOSITY CORRECTION FACTORS

	Viscosity (cP)								
	10	50	100	200	400	600	800	1000	2000
Air unlined baskets, with or without picated inserts	0.65	0.85	1.00	1.10	1.20	1.40	1.50	1.60	1.80
40-mesh lined	0.73	0.95	1.20	1.40	1.50	1.80	1.90	2.00	2.30
60-mesh lined	0.77	1.00	1.30	1.60	1.70	2.10	2.20	2.30	2.80
80-mesh lined	0.93	1.20	1.50	1.90	2.10	2.40	2.60	2.80	3.50
100-mesh lined	1.00	1.30	1.60	2.20	2.40	2.70	3.00	3.30	4.40

Figure 24. *Pressure drop versus flow rate relation fro basket strainers.*

During the filtration cycle, sludge is fed at approximately 100 psi into each chamber through an inlet pipe in the bottom portion of the filter plate. The number of chambers can range from a few dozen to more than 100. The sludge feed pump continues to feed sludge into the chamber until a predetermined filtering time has been achieved. Filtrate passes onto the cloth on both sides of the chamber. The filtration cycle is completed independently in each chamber. Short filtration cycles produce cake thicknesses of 0.5 to 0.75 in. (12.7-19.1 mm). Once the filtration cycle is complete, the sludge pump is stopped and a diaphragm in the chamber is expanded by water pressurized up to 250 psi. This compresses the sludge on both sides of the chamber into a thin, uniform cake with a solids content of more than 35%.

PART A (Figure 25) - FILTRATION

DIAPHRAGM

PLATE WITH
DIAPHRAGM
PLATE

FILTER CLOTH

CLOTH
SUSPENSION

GROMMET

FILTRATE

SLUDGE

PART B - SQUEEZING

SQUEEZING WATER

FILTRATE

PART C (Figure 25) - CAKE DISCHARGE

VIBRATING SHOE

CAKE

PART D - CLOTH WASHING

WASHING CYLINDER

WASHING NOZZLE
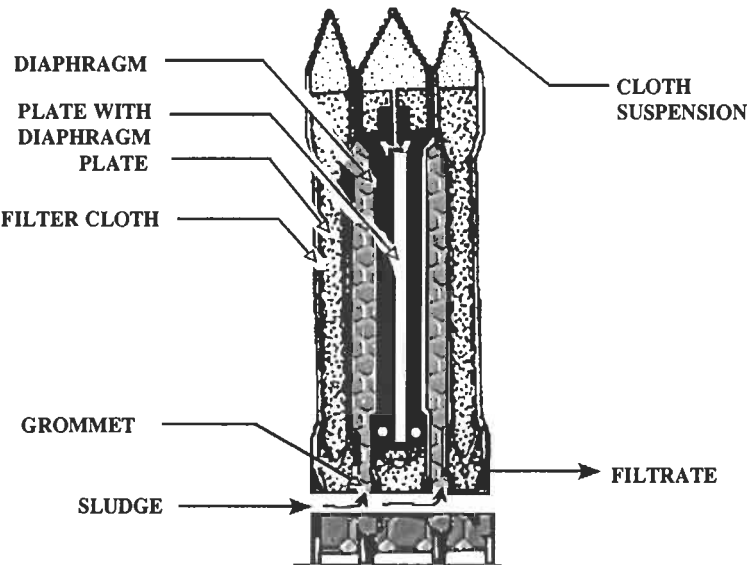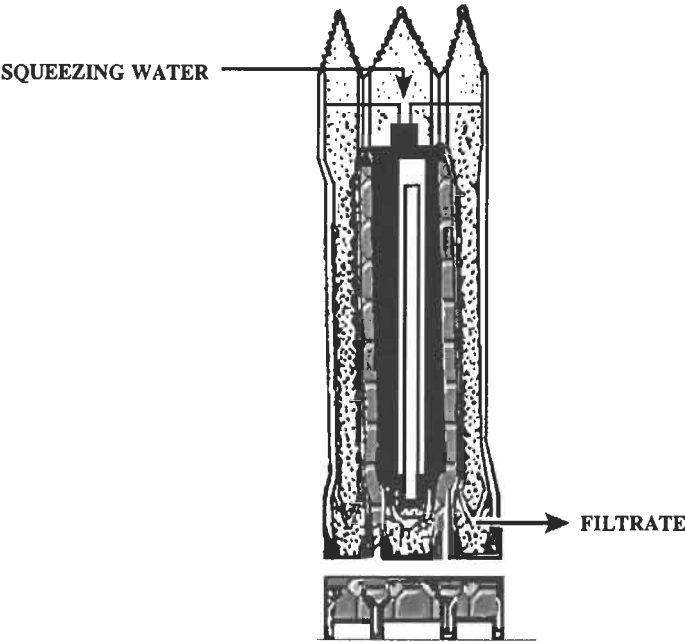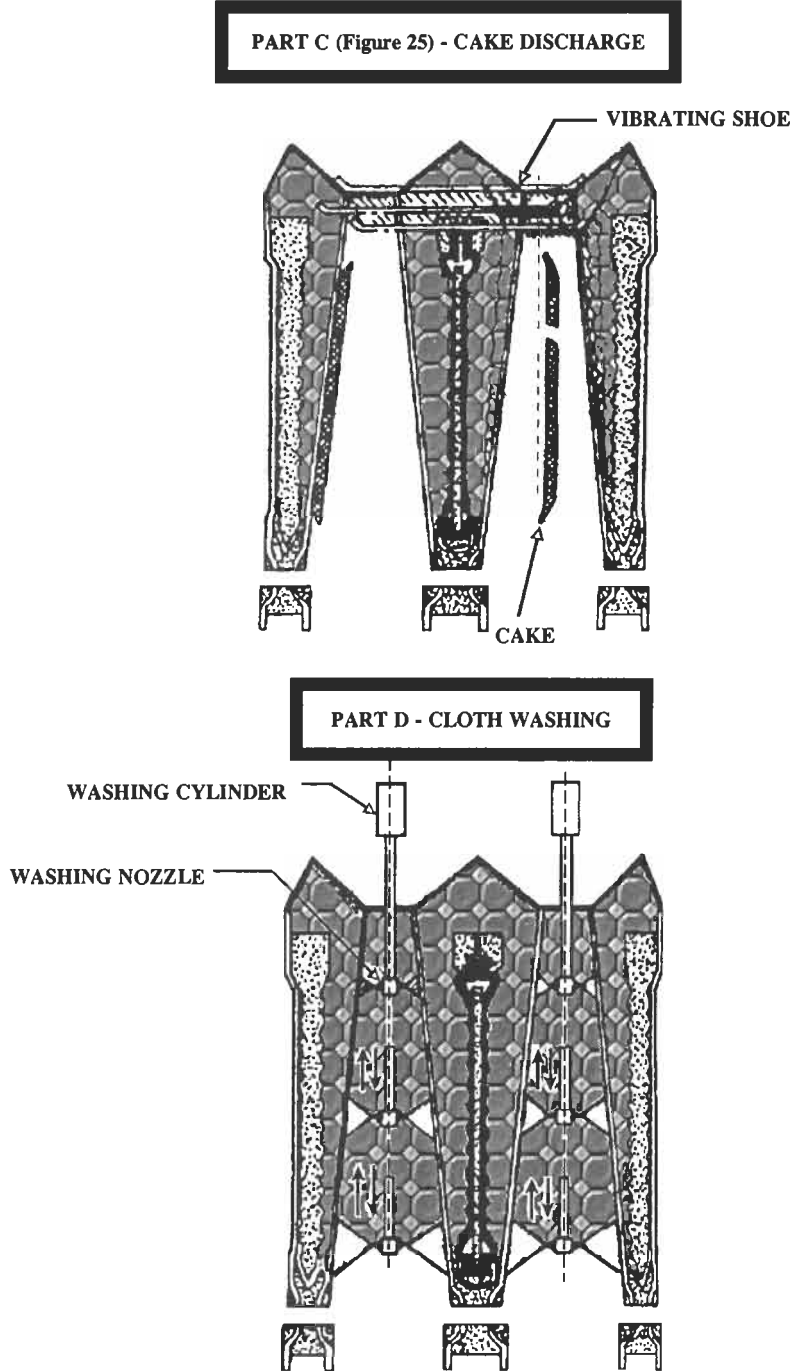
Figure 25. *Diaphragm press operating cycles.*

The uniform water content of the thin cake (no wet cores) results in easier shredding and conveying and makes it much more adaptable to self-sustained thermal destruction or landfill. Optimum filtering and squeezing time cycles vary, depending on the type of sludge, and can be determined accurately by bench tests. The squeezing water is recycled. A hydraulic ram keeps the chambers in position during both cycles. On completion of the filtration and squeeze cycles, the chambers are automatically opened and the cakes are discharged, usually onto a belt conveyor. No precoating is required. Two chambers are normally opened at a time in sequence. This reduces the impact loading on the belt conveyor. Any sludge or filtrate remaining in the feed and filtrate lines is automatically purged by high-pressure (100-psi) air before the next cycle begins. The purging prevents wet sludge from discharging and keeps sludge lines from plugging.Cake discharge from filter presses is a relatively fast step. After a number of cycles (depending on the sludge type), the filter cloth will require cleaning. This can be accomplished manually or can be performed automatically at preset frequencies with an automatic cloth washer using a jet of 1,000-psi wash water. Where even faster cake discharge is desired or where sludge cakes may tend to be sticky, automatic cloth vibrators can be provided. These units help speed mechanical discharge and help remove cakes where poor sludge conditioning causes excessive sticking. This reduces the need for continuous monitoring by operators. Cloth vibrators also simplify cloth selection, since cloths can be selected to assure clearer filtrate or better filtering qualities rather than sacrificing these advantages for a cloth that allows for filter discharge characteristics. The process of cake discharging is illustrated in Figure 26.
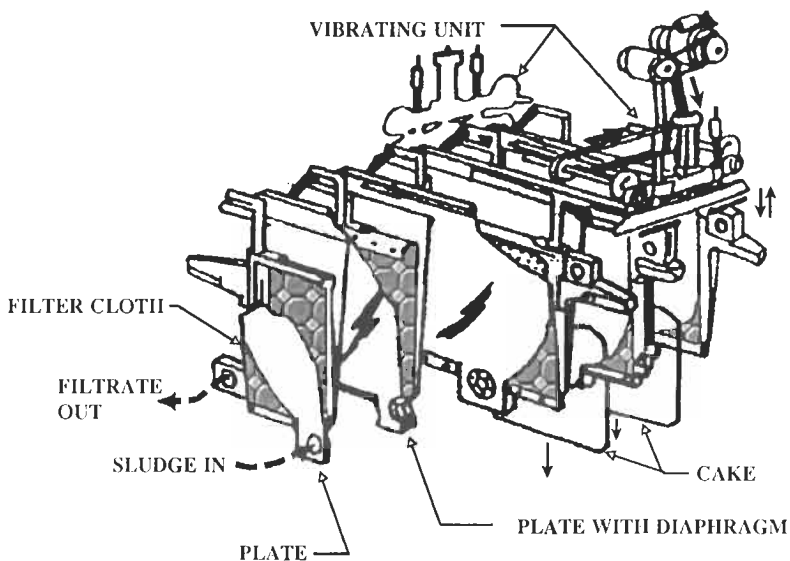
Figure 26. *Cake discharge from a diaphragm filter press.*
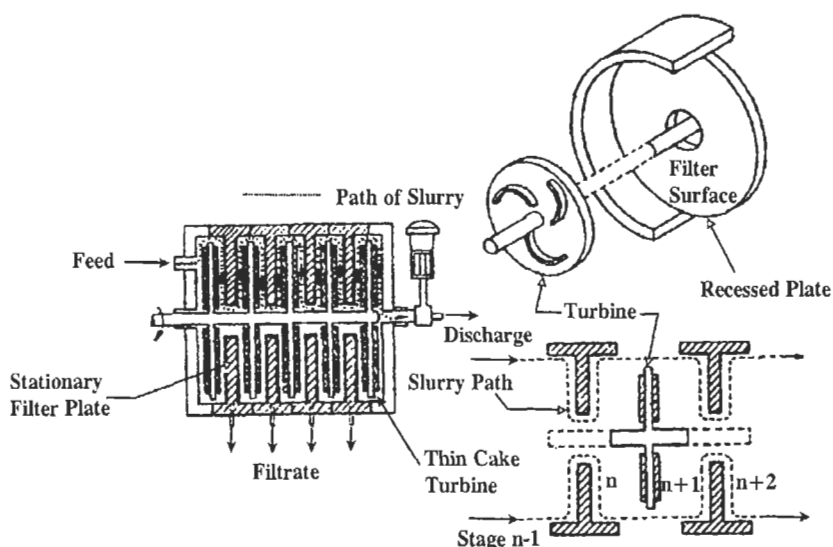
Figure 27. *Components of a staged, thin-film filter/thickener.*

High Pressure, Thin Cake Filters: Thin-cake staged filters have been used effectively at high flowrates per unit area for many years. Use of ultra-thin cakes is a useful technique for increasing flowrates. The basic elements of the filter are illustrated in Figure 27. Filter surfaces are recessed plates equipped with rotating turbines that maintain permanent precoat-type thin cakes throughout the filter. Cake thickness is prevented from growing beyond the in situ precoat formed during the first few minutes of the operation by blades on a rotating shaft passing through the axis of the filter. Slurry flows into the first stage and then flows around the turbines and through the clearances between the shaft and active filter surfaces. As liquid is removed, the thickening slurry moves from stage to stage. The unit acts as a filter-thickener, producing a continuous discharge that may contain a higher solids content than is normally encountered in conventional filters. The turbine plates sweep close to the filter cloths, leaving a thin, permanent cake on each stationary plate. Even in the last stage of the filter, where the slurry is highly non-Newtonian, a thin, easily identifiable, hard cake is maintained. At low turbine velocities, the blades serve as scrapers that limit the cake thickness to be dimensions of the clearance. At higher velocities, the cake thickness is reduced and can be as thin as 1.0 mm with 3-mm clearance. For filters that depend entirely on fluid action, shear forces at the cake surface depend on fluid properties and velocity distributions. The combination of high pressure (300 psi) and thin cakes produces high rates. Washing is accomplished either cocurrently or countercurrently. Separate filters in series can be employed in a manner similar to conventional thickeners. Washing may also be

performed within a single unit, whereby, an initial portion of the filter must be used to remove liquid. In this case, the final stages are used for concentration. Clean wash liquid may be injected after the initial filtering at one or several intermediate stations. Injection wash tends to increase the overall filtrate rate but decreases the cake output rate.

Centrifugal Filtration: Filtering centrifuges are distinguished from standard centrifugation by a filtering medium incorporated into the design. Slurry is fed to a rotating basket or bowl having a slotted or perforated wall covered with a filtering medium such as canvas or metal- reinforced cloth. The angular acceleration produces a pressure that transports the liquor through the filtering medium, leaving the solids deposited on the filter medium surface as a cake. When the feed stream is stopped and the cake spun for a short time, residual liquid retained by the solids drains off. This results in final solids that are considerably drier than those obtained from a filter press or vacuum filter. Principal types of filtering centrifuges are suspended batch machines, automatic short-cycle batch machines and continuous conveyor centrifuges. In suspended centrifuges, the filter medium is usually canvas or a similar fabric, or woven metal cloth. Automatic machines employ fine metal screens. The filter medium in conveyor centrifuges is usually the slotted wall of the bowl itself. Figure 28 provides an example of this machine. The system combines the features of a centrifuge and a screen. Feed enters the unit at the top and is immediately brought up to speed and distributed outward to the screen surface by a set of vanes. Water or other liquid is forced by the sudden centrifugal action through the screen openings into an effluent housing.
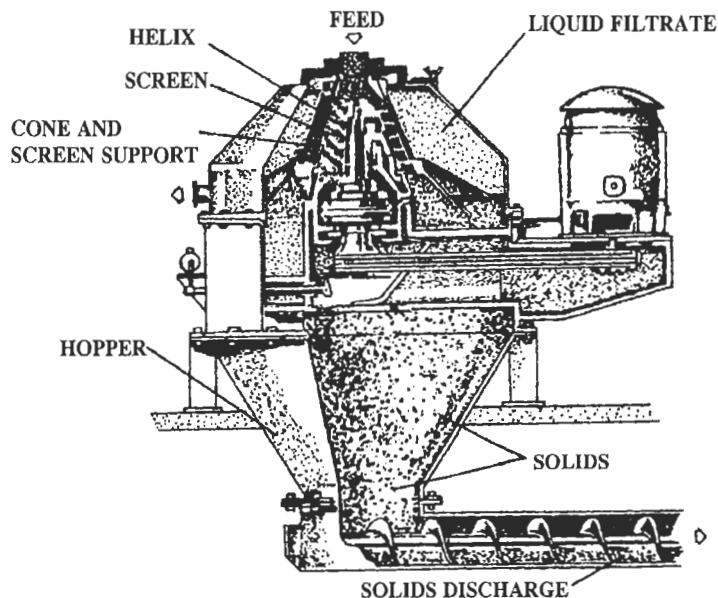
Figure 28. *Cutaway view of filter centrifuge.*

As solids accumulate, they are gently moved down the screen by the slightly faster rotating helix. With the increase in screen diameter, higher centrifugal gravities are encountered and solids are dispersed over a gradually increasing area, thus forming a thin, compact cake from which the remaining liquid is extracted. The relatively dry solids are blown out the bottom of the rotor by a set of vanes into a conical collection hopper.

Rapid Filteration: Rapid filtration is a standard unit operation in wastewater treatment. The operation is performed either in open gravitational flow filters or in closed pressure filters. Rapid pressure filters have the advantage of being able to be inserted in the pumping system, thus allowing use of a higher effective loadings. Note that pressure filters are not subject to development of negative pressure in a lower layer of the filter. These filters generally support higher speeds, as the available pressure allows a more rapid flow through the porous medium made up by the filter sand. Pressure filtration is generally less efficient than the rapid open type with free-flow filtration. Pressure filters have the following disadvantages. The injection of reagents is complicated, and it is more complicated to check the efficiency of backwashing. Work on the filter mass is difficult considering the assembly and disassembly required. Also, the risk of breakthrough by suction increases. Another disadvantages is that pressure filters need a longer filtration cycle, due to a high loss of head available to overcome clogging of the filter bed. Another option is to use open filters, which are generally constructed in concrete. They are normally rectangular in configuration. The filter mass is posed on a filter bottom, provided with its own drainage system, including bores that are needed for the flow of filtered water as well as for countercurrent washing with water or air. There are several types of washing bottoms. One type consists of porous plates which directly support the filter sand, generally without a layer of support gravel. Even if the system has the advantage of being of simple construction, it nevertheless suffers from incrustation. This is the case for softened water or water containing manganese. Porous filters bottoms are also subject to erosion or disintegration upon the filtration of aggressive water. The filter bottom is often comprised of pipes provided with perforations that are turned toward the underpart of the filter bottom and embedded in gravel. The lower layers are made up of gravel of approximate diameter 35 - 40 mm, decreasing up to 3 mm. The filter sand layer, located above this gravel layer, serves as a support and equalization zone. Several systems of filter bottoms comprise perforated self-supporting bottoms or false bottoms laid on a supporting basement layer. The former constitutes a series of glazed tiles, which includes bores above which are a series of gravels in successive layers. All these systems are surpassed to some extent by filter bottoms in concrete provided with strainers. The choice of strainers should in part be based on the dimensions of the slits that make it possible to stop the filter sand, which is selected as a function of the filtration goal. Obstruction or clogging occurs only rarely and strainers are sometimes used. Strainers may be of the type with an end that continues under the

filter bottom. These do promote the formation of an air space for backwashing with air. If this air space is not formed, it can be replaced by a system of pipes that provide for an equal distribution of the washing fluids. Pressure filters are worth noting. These are usually set up in the form of steel cylinders positioned vertically. Another variation consists of using horizontal filtration groups. This has the drawback that the surface loading is variable in the different layers of the filter bed; moreover, it increases with greater penetration in the filter bed (the infiltration velocity is lowest at the level of the horizontal diameter of the cylinder). The filter bottom usually consists of a number of screens or mesh sieves that decrease in size from top to bottom or, as an alternative, perforated plates supporting gravel similar to that used in the filter bottoms of an open filter system. Filter mass washing can influence the quality of water being filtered. Changes may be consequent to fermentation, agglomeration, or formation of preferential channels liable to occur if backwashing is inadequate. Backwashing requires locating a source that will supply the necessary flow and pressure of wash water. This water can be provided either by a reservoir at a higher location or by a pumping station that pumps treated water. Sometimes an automated system is employed with washing by priming of a partial siphon pumping out the treated water stored in the filter itself. An example is shown in Figure 29. The wash water must have sufficient pressure to assure the necessary flow.
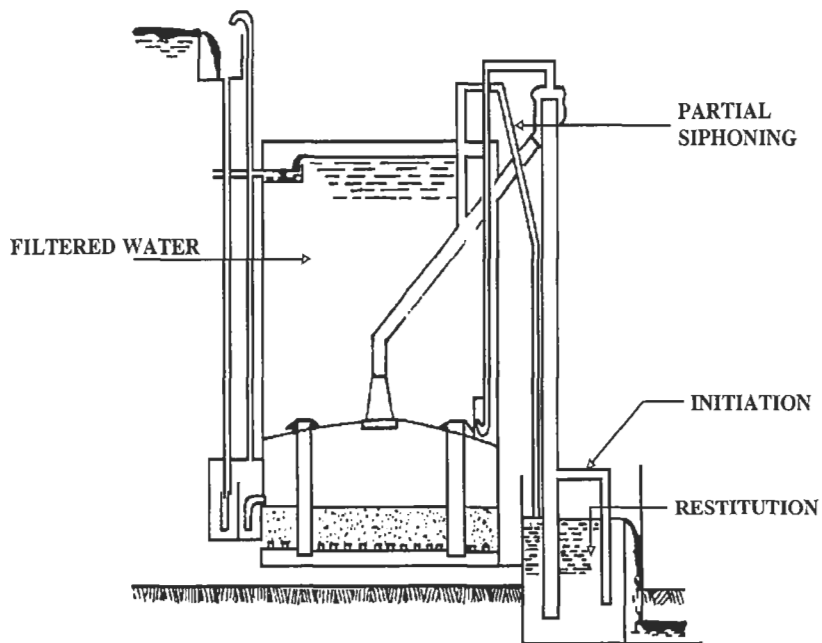
Figure 29. *Automatic backwashing filter with a partial siphon.*

Washing of the filter sands is accomplished followed by washing with water and in most cases including a short intermediate phase of simultaneous washing with air and water. The formation of a superficial crust on the filter sand is avoided by washing with air. After washing with air, water flow is gradually superimposed on the air flow. This operational phase ends at the same time that the wash air is terminated, to avoid the filter mass being blown away. The wash water contains materials that eventually require treatment in a sludge treatment plant. Their concentration varies as a function of the washing cycle. Accounting for the superficial load in filtration, velocity of the wash water, and length of the filtration cycle, it may be assumed that the water used for washing will not attain 5 % of the total production. For new installations the first washing cycles result in the removal of fine sand as well as all the other materials usually undesirable in the filter mass, such as particles of bitumen on the inner surface of the water inlet or other residuals from the crushing or straining devices of the filter media. Consequently, it is normal that at the beginning of operation of a filter sand installation, dark colored deposits appear at the surface of the filter mass. In the long term they have no consequence and disappear after a few filtration and wash cycles. If, after several weeks of filtration, these phenomena have not disappeared, it will be necessary to examine the filter sand. The elimination of fine sand must stop after 1 or 2 months of activity. If this sand continues to be carried away after the first several dozen washes it is necessary to reexamine the hydraulic criteria of the washing conditions, the granulometry of the filter mass, and the filter's resistance to shear and abrasion.

Theory and Design Methodology

In this subsection, basic design theory for preliminary sizing and specifying equipment are reviewed. Some sample design calculations are included. References cited at the end of the chapter can be consulted for more detailed information and design methods. For solid-liquid separation methods, the reader should refer to *Liquid Filtration*, 2nd edition, by N. P. Cheremisinoff, Butterworth-Heinemann Publishers (1998).

Gas Filtration: Vendors are the primary source for sizing baghouses, however the process engineer should at the very least make an estimate of the pressure drop requirements for an intended system. The pressure drop will, of course, provide information needed on the approximate size of the fan needed, and the energy requirements needed to operate a system. In fact, pressure drop is the primary parameter in establishing particle removal efficiency for a fabric filtration unit, and hence, is usually the starting place for discussions with the equipment supplier.

There are conflicting procedures for predicting pressure drop across fibrous filters. The three chief methods of predicting pressure drop are based on the hydraulic

radius theory, drag theory, and dimensional analysis. Experimental studies have shown dimensional analysis to be the most reliable, and it yields an equation for accurate pressure drop prediction. It is based on dimensional analysis of Darcy's law of flow through porous media and relates the pressure drop to the filter porosity. Darcy's original equation states:

$$\Delta PA / hq' = kw$$

where: A = cross-sectional area of filter

ΔP = pressure drop

h = filter thickness

q' = volumetric flowrate

k = permeability of the medium

w = fluid (gas) velocity

Darcy's empirical equations assume the gas to behave ideally; however, a modification of this is the following expression:

$$\Delta PA/hq' = k_3 w s_0^2 (1 - \epsilon)^2/\epsilon^3$$

where: k_3 = constant

s_0 = surface area/unit volume of solid material

ϵ = cloth porosity

From dimensional analysis, the following empirical expression is obtained:

$$k = \Delta PA d_e^2/hq'w = 64 (1 - \epsilon)^{1.5} (1 + 56 (1 - \epsilon)^3)$$

where: k = permeability coefficient

d_e = effective fiber diameter

This formula has proven accurate for fiber diameters ranging from 1.6 to 80 μ, and for filter porosities ranging from 0.700 to 0.994.

Another empirical expression that can be used to predict pressure drop, again based upon application of dimensional analysis is given by the following formula. This relationship is applicable to fabrics having porosities over the range of 0.88 to 0.96,

and for fiber diameters ranging from 0.1 to 0.3 μ. In this formula the parameters are defined as follows: λ is the mean-square fiber diameter and k" is the resistance coefficient.

$$\Delta PA\lambda/hq'w = k"(1 - \epsilon)^{1.5}$$

A final empirical expression that can be used is given below. This equation relates pressure drop to fabric properties which are readily obtainable from equipment suppliers.

$$\Delta P = w\xi(k_0 + k_1W_A)$$

where: ξ = air to cloth ratio

k_0 = weave resistance coefficient

k_1 = cake resistance coefficient

W_A = weight of filtrate cake per unit area of surface

Solid-Liquid Filtration: As already described, solid-liquid filtration operations handle sludges of varying characteristics ranging from granular, incompressible, free-filtering materials to slimes and colloidal suspensions that are compressible and tend to foul the filtering medium. The important characteristics of a sludge are its structure (granular and open or colloidal and dense) and its degree of compressibility. With an incompressible sludge, the resistance of the cake is essentially independent of the pressure. If the sludge is compressible, resistance increases rapidly with increasing pressure. In the process of suspension separation, a continuous accumulation of solids takes place, forming a wet filter *cake* and a *filtrate*. This type of filter operation provides a particle retention that exceeds that obtained based on the hole size of the filtering medium. Smaller particles tend to pass through the medium's pores. In contrast to this type of *filtering medium*, filtration in the depth of the pores of a filtering medium is responsible for capturing particles much smaller than the pore sizes in the actual passages. *Depth filtration* exhibits increased retentivity as particles travel from the upstream face of the filter plate to the downstream side. Fine particles are removed in the deeper portions of the filter, filling the pores. The most common form of filtering in the chemical and process industries is *cake filtration* on the filtering medium. This involves handling the permeability of a bed of porous material. Filtration is based on a complicated interaction between the sludge and the filtering medium. The solid particles entrained in the liquid flow through the filtering medium encounter different conditions. Particles larger than the pores on the filter media surface are captured.

Many particles whose sizes arc less than that of a pore at its narrowest cross section tend to pass through the filter altogether, along will, the filtrate. However, a portion of these particles is captured inside the filter due to adsorption onto the walls of the pores, or by impingement in the course of its twisted flow path. Such an arrested particle decreases the effective flow cross section, while increasing the probability of retaining the particles that follow. Some particles simply become lodged in the pore, making it impenetrable for other particles. With high solids concentration sludges, even particles that are relatively small in comparison to the pore size may not enter into the media and will tend to remain on the filter surface forming "bridges" over individual openings in the filter plate.

Filtrate flows through the filter medium and cake because of an applied pressure, the magnitude of which is proportional to the filtration resistance. This resistance results from the frictional drag of the liquid as it passes through the filter and cake. Hydrostatic pressure varies from a maximum at the point where liquid enters the cake, to zero, where liquid is expelled from the medium; consequently, at any point in the cake the two are complementary. In other words, the sum of hydrostatic and compressive pressures on the solids always equals the total hydrostatic pressure at the face of the cake. As such, the compressive pressure acting on the solids vary from zero at the face of the cake to a maximum at the filter medium. When the space above the sludge is connected to a source of compressed gas, or the space under the filter plate is connected to a vacuum source, filtering is accomplished under a constant pressure differential, because the pressure in the receivers is maintained constant. In this case, the rate of the process decreases due to an increase in the cake thickness and, consequently, its resistance. If the sludge is fed to the filter with a reciprocating pump having constant capacity then filtering takes place under constant flowrate. In this case, the pressure differential increases due to an increase in the cake resistance. If he sludge is fed by a centrifugal pump, its capacity decreases with an increase in cake resistance and the filtering is under variable pressure differences and flowrates.

The most favorable filtration process with cake formation is the process in which there is no clogging of the filtering medium. Such a process is observed at sufficiently high concentrations of solid particles in sludge. This concentration may be assumed conditionally to be in excess of 1% by volume. To prevent pore clogging in the filtering medium when handling relatively low solids concentrations (e.g., 0.1 to 1% by volume), general practice is to increase the solids concentration in thickeners before the sludge is fed to the filter. To improve the filtration characteristics of sludges that are difficult to filter, *filter aids and/or flocculants* often are used to increase particle size. A filter aid is a finely divided solid material, consisting of hard, strong particles that are, in mass, incompressible. The most common filter aids (as an admix to the sludge) are diatomaceous earth, expanded perlite, solkafloc, fly ash or carbon. Filter aids may be applied in one of two ways. The first method involves the use of a precoat filter aid, which can be applied as a

thin layer over the filter before the sludge is pumped to the apparatus. This prevents fine sludge particles from becoming so entangled in the filter plate that the resistance of the filtering medium itself becomes high. Furthermore, it facilitates the removal of the cake at the end of the filtration cycle. The second application method involves the incorporation of a certain amount of the material with the sludge before introducing it to the filter. The addition of filter aids increases the porosity of the sludge, decreases its compressibility and reduces the resistance of the cake. An acceptable filter aid should have a much lower filtration resistance than the material with which it is being mixed.

It should also reduce the filtration resistance by two-thirds to three-fourths with the addition of no more than 25% by weight of filter aid as a fraction of total solids. The addition of only a small amount of filter aid (e.g., 5% of the sludge solids) can cause a significant increase in filtration resistance. When the amount of filter aid is so small that the particles do not interact, they form a coherent structure and resistance may be affected adversely. An increase in the particle size tends to improve the filtration process, which may be achieved by *flocculation*. Flocculants are filtered out more readily into a cake, which is permeable and does not clog the filtering medium. Flocculation is an application of the principles of colloid chemistry. Only when conditions are proper will particle flocs form. Mild agitation assists in bringing particles into contact for the action to take place. Many dispersions are often unstable and chemically ready to flocculate. Others require that the dispersing agent on their surfaces be neutralized or precipitated. There are others in which the particulate matter need to be flocculated with a precipitate (e.g., in lime-alum clarification of wastewater). Once chemical conditions are satisfied, flocculation action proceeds. The operation of chemical pretreatment consists of flocculation or coagulation, which involves the manipulation of charges on the particulates. Small particulates, which are highly charged, form stable suspensions that neither readily settle nor filter.

The addition of trivalent cationic salts (e.g., $AlCl_3$, $FeCl_3$) or a change in the solution pH are common methods employed for changing the surface potential. Most natural substances are negatively charged. The addition of coagulants, in the form of salts, acids or bases, tends to neutralize surface charges, thus permitting attractive van der Waals-London-type forces to promote particle agglomeration. High-molecular weight linear polymers (which may be cationic, anionic or nonionic) combine with charged particulates to form large sedimenting flocs. Bentonite and kaolin clays are used principally as extender-aids in combination with flocculants. Because of active sites present on the extenders, they attach to nucleating sites. Their overall effect is to increase the density of the flocs and settling rates.

Filtration frequently is accompanied by hindered or free gravitational sedimentation of solid particles. The directions of action of gravity force and filtrate motion may be cocurrent, countercurrent or cross current, depending on the orientation of the

filtering plate, as well as the sludge location over or below the filtering plate. Particle sedimentation complicates the filtration process and influences the controlling mechanisms. Furthermore, these influences vary depending on the relative directions of gravity force and filtrate motion.

If the sludge is above the filter plate, particle settling leads to more rapid cake formation with a clear filtrate, which can be evacuated from the filter by decanting. If the sludge is under the filter plate, particle settling will prevent cake formation, and it is necessary to mix the sludge to maintain its homogeneity. When the cake structure is composed of particles that are readily deformed or become rearranged under pressure, the structures generally are termed compressible cakes. Those not readily deformed are called semicompressible, and those that deform only slightly are considered noncompressible. Porosity (the ratio of pore volume to volume of cake) does not decrease with increasing pressure differential. The porosity of a compressible cake decreases under pressure, and its hydraulic resistance to the flow of the liquid phase increases with pressure differential across the filter media. Cakes containing particles of inorganic substances, with sizes in excess of 100 μ, may be considered practically incompressible. Examples are sand particles and crystals of carbonates of calcium and sodium. The cakes containing particles of metal hydroxides, such as ferric hydroxide, cupric hydroxide and aluminum hydroxide, as well as sediments consisting of easily degradable aggregates, which are formed from primary fine crystals, are compressible. After completion of cake formation, treatment of the cake is dependent on the specific filtration objectives. For example, the cake itself may have no value, whereas the filtrate may.

Depending upon the disposal method and properties of the particles, the cake may be discarded in a dry form or as a slurry. In both cases, the cake is subjected to washing, following its formation, either immediately, or after a period of drying if the volume of wash liquor is limited, or to avoid filtrate dilution or excessive wash concentrations. In some cases a second washing is required, followed by a drying period in which filtrate must be removed from the cake; or wet discharge follows for disposal; or repulping and a second filtration occur; or dry cake disposal is preferable. Similar treatment options are employed in cases where the cake is valuable and all contaminating liquors must be removed, or in which both cake and filtrate are valuable. In the latter, cake-forming filtration is employed without washing, to dewater cakes in which a contaminating liquor forms the moisture content of the cake. Two basic methods of cake washing are displacement and dilution. Displacement involves pouring the washing liquid onto the cake surface in the form of a liquid layer or by means of atomization. The liquid passes through the pores , thereby displacing bound liquid in its path. The method is used with easily washed cakes that have no blind pores. In the dilution method, the cake is removed from the filter and mixed with the liquid. The suspension is then separated on the filter. This method is preferred with cakes that are difficult to wash.Design formulations developed for a filtration process analysis are based upon a two part

concept or approach. The first half consider flow within the cake, while the second examines the flows imposed on the cake and pumping system. The characteristics of the pump relate the pressure and the flowrate at the exit face of the cake. It is the cake resistance that determines the pressure drop. During filtration, liquid flows through the porous filter cake in the direction of a decreasing hydraulic pressure gradient. The porosity, ϵ, is at a minimum at the point of contact between the cake and filter plate, i.e., where $x = 0$, and it is a maximum at the cake surface ($x = L$) where the sludge enters. The drag imposed on each particle is transmitted to adjacent particles. Therefore, the net solid compressive pressure increases as the filter plate is approached, resulting in a decrease in porosity. It may be assumed that particles are in contact at one point only on their surface, and that liquid surrounds each particle completely. Hence, the liquid pressure acts uniformly in a direction along a plane perpendicular to the direction of flow. As the liquid flows past each particle, the integral of the normal component of force leads to the form drag, and the integration of the tangential components results in the frictional drag. If the particles are nonspherical, we still may assume single point contacts between adjacent particles.

We consider flow through a cake with the membrane located at a distance, x, from the filter plate. Neglecting all forces in the cake other than those created by drag and hydraulic pressure, a balance from x to L gives:

$$F_s + AP_L = AP$$

where the applied pressure, P, will be a function of time but not of the distance, x. The term, F_s, is the cumulative drag on particles, increasing in the direction from $x = L$ to $x = 0$. As single-point contact is assumed, the hydraulic pressure, P_L, is effective over the entire cross section (A) of the cake. Dividing the above expression by A and denoting the compressive drag pressure by $P_s = F/A$, we obtain:

$$P_L + P_s = P$$

The term P_s is a fictitious pressure because the cross-sectional area, A, is not equal to either the surface area of the particles or the actual contact areas. In actual cakes, there is a small area of contact, A_c, and the solid pressure may be defined as F_s/A_c. Taking differentials with respect to x in the interior of the cake, we obtain:

$$dP_s + dP_L = 0$$
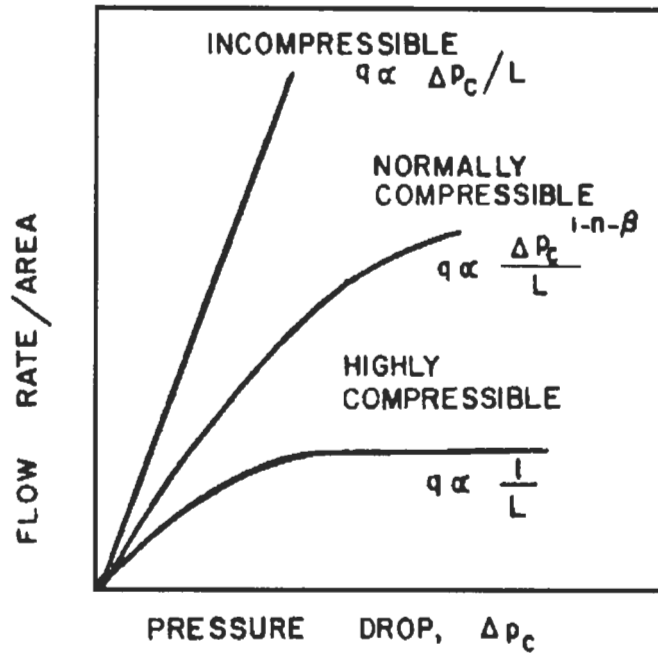
Figure. 30. *Flowrate - pressure drop relationships.*

This means that drag pressure increases and hydraulic pressure decreases as fluid moves from the cake's outer surface toward the filter plate. From Darcy's law, the hydraulic pressure gradient is linear through the cake if the porosity, ϵ, and specific resistance, α, are constant. The cake then may be considered "incompressible." This is shown by the straight line on the plot of flowrate per unit filter area versus pressure drop in Figure 30. The variations of porosity and specific resistance are accompanied by varying degrees of compressibility, also shown in Figure 30.

Because pore sizes in the cake and filter plate are small and the liquid velocity through the pores is low, the flow of filtrate may be considered laminar and Poiseuille's law is applicable. Filtration rate is directly proportional to the difference in pressure and inversely proportional to the fluid viscosity, as well as to the hydraulic resistance of the cake and filter plate. Because the pressure and hydraulic resistances of the cake and filter plate change with time, the variable rate of filtration may be expressed as:

$$u = dV/Ad\tau$$

where V = volume of filtrate, A = filtration area (m^2), and τ = filtration time (s).

Assuming laminar flow through the filter channels, the basic differential equation of filtration is simply stated as follows:

$$u = dV/Ad\tau = \Delta P/[\mu(R_c + R_f)]$$

where ΔP = pressure difference (N/m^2), μ = viscosity of filtrate ($N\text{-}s/m^2$), R_c = filter cake resistance (liter/min), R_f = initial filter resistance (resistance of filter plate and filter channels, liter/min), u = filtration rate (m/s) (filtrate flow through cake and filter plate), and $dV/d\tau$ = filtration rate (m^3/s) filtrate flow. The value of R_f may be assumed constant during filtering. This assumption is based on any increase due to solids penetrating inside the pores. Filter cake resistance, R_c, is the resistance to filtrate flow per square meter of filtration area. R_c increases with cake thickness during filtration. At any instant, R_c depends on the mass of solids deposited on the filter plate as a result of the passage of V m^3 of filtrate. To determine the relationship and residence time, τ, the above expression must be integrated. This enables R_c to be expressed in terms of V.

Defining the ratio of cake volume to filtrate volume as x_o, then the cake volume is $x_o V$. The cake volume also may be expressed by the product $h_c A$, where h_c is the cake height in m. Hence,

$$x_o V = h_c A$$

Hence, the thickness of the cake, uniformly distributed over the filter plate, is

$$hc = x_o(V/A)$$

And the filter cake resistance may be expressed as

$$R_c = r_o x_o (V/A)$$

where r_o is the specific volumetric cake resistance, m^{-2}. As follows from this expression, r_o characterizes the resistance to the liquid by a cake of 1-m thickness. Substituting for R_c into this expression, we obtain:

$$dV/Ad\tau = u = \Delta P/[\mu\{r_o x_o(V/A) + R_f]$$

Parameter x_o can be expressed in terms of the ratio of the mass of solid particles settled on the filter plate to the filtrate volume, x_w, and, instead of r_o, a specific mass cake resistance, r_w, is used. That is, r_w is the resistance to the flow presented by a uniformly distributed cake in the amount of 1 kg/m². Replacing units of volume by mass, the term $r_o x_o$ into the above expression changes to $r_w x_w$. Neglecting the filter plate resistance (i.e., $R_f = 0$), then:

$$r_o = \Delta P / \mu h_c u$$

At $\mu = 1$ N-s/m², $h_c = 1$ m and u $= 1$ m/s, the value $r_o = \Delta P$. Thus, the specific cake resistance equals the pressure difference required by the liquid phase (with a viscosity of 1 N-s/m²) to be filtered at a rate u $= 1$ m/s for a cake 1 m thick. This hypothetical pressure difference is, however, beyond a practical range. For highly compressible cakes, the value ro reaches 10^{12} m⁻² or more. Assuming V $= 0$ (at the start of filtration) where there is no cake over the filter plate, the equation becomes:

$$R_f = \Delta P / \mu u$$

At $\mu = 1$ N-s/m² and u $= 1$ m/s, $R_f = \Delta P$. This means that the filter plate resistance is equal to the pressure difference necessary for the liquid phase to pass through the filter plate at a rate of 1 m/s. For many filter plates, R_f is typically 10^{10} m⁻¹.

With the above as an introduction, we now consider the important operational case of filtration performed under constant pressure. In practice, all the parameters defined above are nearly constant under steady state conditions except V and τ, which are varied by the operator. We may therefore integrate the working expression for filtration over the limits of volume from 0 to V, and for residence time over the limits of 0 to τ:

$$\int \mu (r_o x_o (V/A) + R_f) dV = \int \Delta P A d\tau$$

Upon integration and reworking the relationship (details may be obtained from *Fluid Mechanics and Unit Operations*, D. S. Azbel and N. P. Cheremisinoff, Ann Arbor Science Publishers, Mich. (1983)), we obtain the following working formula:

$$V^2 + 2(R_f A / r_o x_o) V = (2 \Delta P A^2 / \mu r_o x_o) \tau$$

This expression shows the relationship between filtration time and filtrate volume. The equation is applicable to both incompressible or compressible cakes, because at constant ΔP the values r_o and x_o are constant. For constant ΔP, an increase in the filtrate volume results in a reduction in the filtration rate. If we assume a definite filtering apparatus and set up a constant temperature and filtration pressure, then the values of R_f, r_o, μ and ΔP will be constant. We now take note of the well-known filtration constants K and C, which are derived from the above expressions:

$$K = 2\Delta PA^2 / \mu r_o x_o$$
$$C = R_f A / r_o x_o$$

The filtration process may therefore be expressed by a very simple relationship:

$$V^2 + 2VC = K\tau$$

Constants K and C can be readily obtained from experiments conducted on a prototype machine, from whence the volume of filtrate obtained for a definite time interval (for a specified filter, at the same pressure and temperature) can be calculated. If process parameters are varied, new constants K and C can be estimated from the above expressions. The last expression can be further modified by denoting the constant τ_o as $= C^2/K$, and substituting:

$$(V + C)^2 = K(\tau + \tau_o)$$

This equation is then the expression describing the process of filtration under constant pressure. The form of the relationship is parabolic. From a plot of $(V + C)$ versus $(\tau + \tau_o)$, the filtration process may be represented by a parabola with its apex at the origin, as shown in Figure 31. Moving along the axes to point C and τ_o provides the characteristic filtration curve for the system in terms of volume versus time. Because the parabola's apex is not located at the origin of this new system, it becomes clear why the filtration rate at the beginning of the process will have a finite value, as seen in actual practice. It is often overlooked that constants C and τ_o have physical significance. They are equivalent to a fictitious layer of cake of equivalent resistance. The formation of this fictitious cake follows the same parabolic relationship. Parameter τ_o denotes the time required for the formation of this fictitious cake, and C is the volume of filtrate required. Upon integrating the filtration expression and rearranging terms, a more convenient linear form of the expression is derived, lending itself to a graphical analysis.
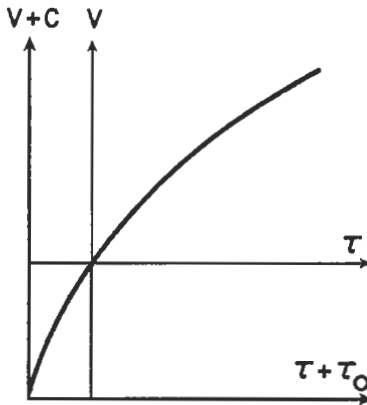
Figure 31. *The typical filtration curve.*   **Figure 32.** *Linear form of equation.*
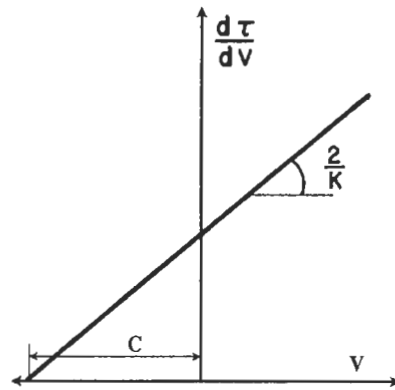
The linear analytical expression is:

$$d\tau/dV = 2V/K + 2C/K$$

A plot of this expression is provided in Figure 32, where the slope of the straight line is 2/K, and the intercept value is C. Experimental determination of $d\tau/dV$ is straightforward. Filtrate volumes V_1 and V_2 are measured at time intervals τ_1 and $\tau2$, respectively. From the linear equation of filtration, the quotient $(\tau_2 - \tau_1)/(V_2 - V_1)$ is:

$$(\tau_2 - \tau_1)/(V_2 - V_1) = (2/K)[(V_1 + V_2)/2] + 2C/K$$

Constants C and K can be determined from several measurements of filtrate volumes taken at different time intervals. There are some doubts as to the actual constancy of C and K during constant pressure filtration. Constants C and K depend on r_o (specific volumetric cake resistance), which, in turn, depends on the pressure drop across the cake. This ΔP causes some changes in the cake, especially during the initial stages of filtration. When the cake is very thin, the main portion of the total pressure drop is exerted on the filter medium. As the cake becomes thicker, the pressure drop through the cake increases rapidly but then levels off to a constant value. Isobaric filtration shows insignificant deviation from the expressions developed. For approximate calculations, it is possible to neglect the resistance of the filter plate, provided the cake is not too thin. Then the filter plate resistance, R_f, is equal to zero, C = 0, and r_o = 0. Hence, a simplified equation is: $V^2 = K\tau$.

The following example helps to illustrate the use of the equations presented up to this point. *An aqueous slurry was filtered in a small laboratory filter press with a pressure drop of 0.5 atm and at a temperature of 20 °C. After 10 minutes, 4.7 liters of filtrate were obtained; after 20 minutes, 7.0 liters were collected. From experiments at other pressures, it was determined that the cake compression coefficient was s = 0.4. We wish to determine the volume of filtrate expected after 30 minutes from a filter press having a filtering area 10 times greater than the laboratory press if the filtration is to be performed at 1.5 atm pressure. The liquid temperature will be 55 °C. We also wish to determine the rate of filtration at the end of the process.*

The solution to this problem is as follows. Evaluate K and C from the experimental data at $\Delta P = 0.5$ atm. through the use of the expression $V^2 + 2VC = K\tau$. For V = 4.7 liters and $\tau = 10$ min, we obtain: $(4.7)^2 + 2 \times 4.7 \times C = 10K$. For V = 7.0 liters and $\tau = 20$ min, we obtain: $(7.0)^2 + 2 \times 7 \times C = 20K$. Solving, we obtain C = 1.04 and K = 3.18. These coefficients change as a function of the filtration area, temperature and pressure. We note that the following empirical expression holds:

$$r_o = a\Delta P^s$$

where a is a coefficient. Constant C may be written in the following form:

$$C = (R_f/ax_o) \times (A/^{\Delta ps})$$

The term r/ax_o is a constant and independent of filtration conditions. We find that the ratio of constant C_2 at pressure 1.5 atm. to constant C_1 for the process under pressure at 0.5atm. is as follows: $C_2/C_1 = (A_2/A_1) \times (\Delta P_1/\Delta P_2)^s$. Then $A_2/A_1 = 10$ and s = 0.4, and hence, we solve for C_2: $C_2 = 1.04 \times 10(0.5/1.5)^{0.4} = 8.85$. Coefficient K is determined from:

$$K = (2/ax_o) \times \Delta P^{1-s} A^2/\mu;$$
$$K_2/K_1 = (\Delta P_2/\Delta P_1)^{1-s} (A_2/A_1)^2 (\mu_1/\mu_2)$$

The viscosity of water at 20 °C is $\mu_1 = 1$ cp; at 55 °C, it is 0.5 cp. Therefore, $K_2 = 3.18 \times (1.5/0.5)^{1-0.4} \times 100 \times (1/0.5) = 1230$. From the values of K_2 and C_2, we determine $\tau_o = (8.85)^2/1230 = 0.0637$. Applying the linear expression of filtration then: $(V + 8.85)^2 = 1230(\tau + 0.0637)$. For time $\tau = 30$ min, we obtain

volume V in liters: $V = \{1230(30 + 0.064)\}^{1/2} - 8.85 = 183$ liters. The final filtration rate (after 30 minutes) is obtained from $dV/d\tau = K/[2(V + C)] = 1230/[2(183 + 8.85)] = 3.2$ liters/min. The average rate of filtration is: $V/\tau = 183/30 = 6.1$ liters/min.

We now turn attention towards the case of constant-rate filtration. When sludge is fed to a filter by means of a positive displacement pump, the rate of filtration is nearly constant, i.e., $dV/d\tau = $ constant. During constant-rate filtration, pressure increases with cake thickness. As such, the principal filtration variables are pressure ind filtrate volume, or pressure and filtration time. Integrating the filtration equation for a constant-rate process, we find that the derivative $dV/d\tau$ can simply be replaced by V/τ, and we obtain:

$$\Delta P = \mu r_0 x_0 (V^2/A^2\tau) + \mu R_f(V/A\tau)$$

For incompressible cakes, coefficient r_0 is constant and independent of pressure. For compressible cakes ($s \neq 0$) r_0 may be estimated from the expression $r_0 = a\Delta P^s$. Substituting for r_0 into the above relation, we obtain:

$$\Delta P = \mu a x_0 \Delta P^s (V/A\tau)^2 \tau + \mu R_f (V/A\tau)$$

Filtration experiments in a prototype machine at constant pressure or constant rate permit determination of ax_0, as well as s and R_f, for a given sludge and filtering medium. Consequently, it is possible to predict the time required for the pressure drop to reach the desired level for a specified set of operating conditions. In the initial stages of filtration, the filter medium has no cake. Furthermore, ΔP is not zero but has a certain value corresponding to the filter medium resistance for a given rate. This initial condition is:

$$\Delta P_0 = R_f \mu(V/A\tau)$$

For an incompressible cake ($s = 0$), the equation takes the form:

$$\mu a x_0 (V/A\tau)^2 + R_f \mu(V/A\tau) = \Delta P$$

As follows for the filtration of incompressible sediment (at a constant rate), the pressure increases in a direct proportion to time. However, the above equation

shows that pressure increases faster than time. In some cases the resistance of the filter medium may be neglected, especially for thick cakes. Assuming $R_f = 0$, our equation simplifies to the following:

$$\Delta P^{1-s} = \mu x_o a (V/A\tau)^2 \tau$$

An increase in pressure influences not only coefficient r_o, but also the cake's porosity. Cake on the filter plate is compressed; consequently, the additional liquid is squeezed out of the cake. Thus, along with a constant feed of sludge, the flowrate will not be stable and will change somewhat in time. The weight of dry solids in a cake is $W' = x_o'V$, where x_o' is the weight of solids in the cake per unit of filtrate volume. The concentration of solids in the feed sludge is expressed by weight fraction, c. It also is possible to determine experimentally the weight ratio of wet cake to its dry content, m. Hence, a unit weight of sludge contains m × c of wet cake. Denoting γ as the specific weight of filtrate, then $(1 - mc)/\gamma$ is the filtrate volume per unit weight of feed sludge. But this unit contains c amount of solids, and the ratio of solids in the cake to the filtrate volume is defined as:

$$x_o' = c\gamma/(1 - mc)$$

Thus, from sludge concentration c and the weight of a wet cake per kg of dry cake solids, x_o' can be determined. If the suspension is dilute, then c is small and the product of m × c is small. This means that x_o' will be almost equal to c. Since x_o' depends on the product mc, at relatively moderate suspension concentrations this effect will not be great and thus may be neglected. However, for the filtration of concentrated sludges, the mentioned phenomena will play some role, i.e., at constant feed the filtrate changes with time.

Variable-rate, variable-pressure filtration is also an important operating mode for filters. Figure 33 illustrates typical pressure profiles across a filter. This figure shows the distribution of static pressure, P_{st}, in liquid and P along the cake thickness and filter plate: (I, II) are boundaries between the cake and sludge at τ'' and τ'; (III, IV) are boundaries between the cake layers or cake and filter plate at τ' and τ''; (V) is the boundary line between the cake and filter plate or the free surface of the filter plate. Curves (1, 3) represent $P_{st} = f(h_{oc})$ at τ', and curves (2, 4) are $P_{st} = f(h_{oc})$ and $P = f(h_{oc})$ at τ''. The compression force in the cake section is defined as: $P = P_1 - P_{st}$. In this statement P_1 is the pressure acting on the sludge over the entire cake thickness and P_{st} is the static pressure over the same section of cake. Pressure P corresponds to the local specific cake resistance $(r_w)x$. At the sludge-cake interface, $P_{st} = P_1$ and $P = 0$; and for the interface between the cake and filter

plate, $P_{st} = P_{st}'$ and $P = P_1 - P_{st}$. P_{st}' corresponds to the resistance of filter plate, ΔP_f, and is expressed by the equation: $\Delta P_f = \mu R_f W$; where W is defined as the filtration rate (m³/m²-s). Not that ΔP_f is constant during the filtration process, and that P is also the driving force of the process. We consider a small element of solid particles weight in a cake of unit area as $x_w dq$, where q is the filtrate volume from 1 m² of filtering area. The incremental pressure drop for this element is:

$$dP/x_w dq = \mu(r_w)_x W$$

Parameter xw undergoes relatively small changes with P. In practice, and average value can be assumed. Note that W is constant for any section of the cake. The above expression may be integrated over the cake thickness for the limits of $P = 0$ to $P = P_1 - P_{st}$, and from $q = 0$ to $q = q$.

$$q = (1/\mu x_w W)\int dP/(r_w)_x$$
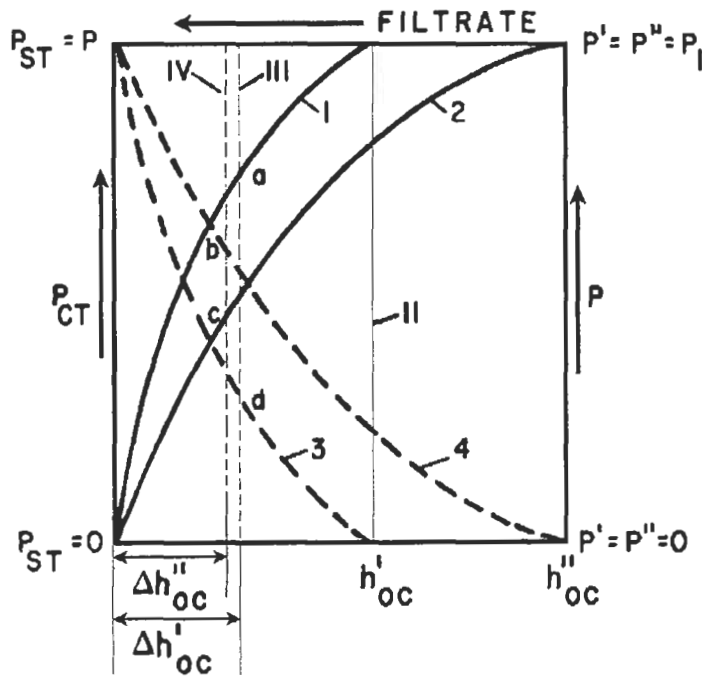
Figure 33. *Pressure profiles across the filter cake and filtrate .*

Parameters q and W are variables when filtration conditions are changed. Coefficient $(r_w)_x$ is a function of pressure; $(r_w)_x = f(P)$. The exact relationship can be derived from experiments in a device called a compression-permeability cell. Once this relationship is defined, the integral of the right hand side of the above equation may be evaluated analytically. Or, if the relationship is in the form of a curve, the evaluation may be made graphically. The interrelation between W and P, is established by the pump characteristics, which define $q = f(W)$ in the integral. Filtration time may then be determined from $dq/d\tau = W$, from which we may state:

$$\tau = \int dq/W$$

The following example illustrates the principles behind variable rate, variable-pressure filtration. *The following data were obtained from separation tests for a particular suspension using a filter: $\mu = 0.00148$ N-s/m^2, $x^w = 3.05$ kg/m^3 and $R_f = 6.56 \times 10^{10\ m-1}$. The relationship $(1/r_w)_x = f(P)$ was obtained from a compression-permeability cell and the results are given by curve 1 in Figure 34. The pump characteristics in terms of capacity per unit filter area are given by curve 1 in Figure 35. Determine the relationship between filtrate volume and the time of filtration.* The solution to this problem is as follows. According to the expression $\Delta P_f = \mu R_f W$, the static pressure at the interface between the cake and filter plate is numerically equal to the plate resistance provided that the pressure exerted on the free surface of the filter plate is equal to zero. Hence: $P_{st}' = \Delta P_f = 0.00148 \times 6.56 \times 10^{10}$ W $= 97.1 \times 10^{10}$ W.
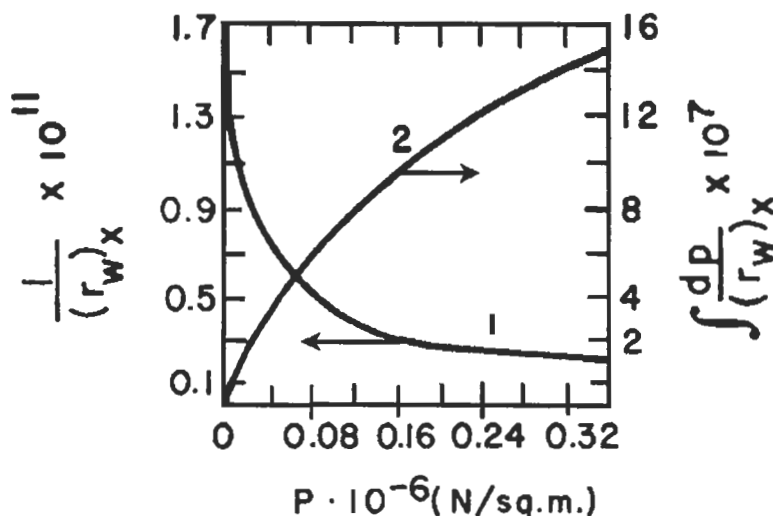
Figure 34. *Specific resistance $1/(r_w)_x$ vs. P (curve 1) and integral (curve 2).*
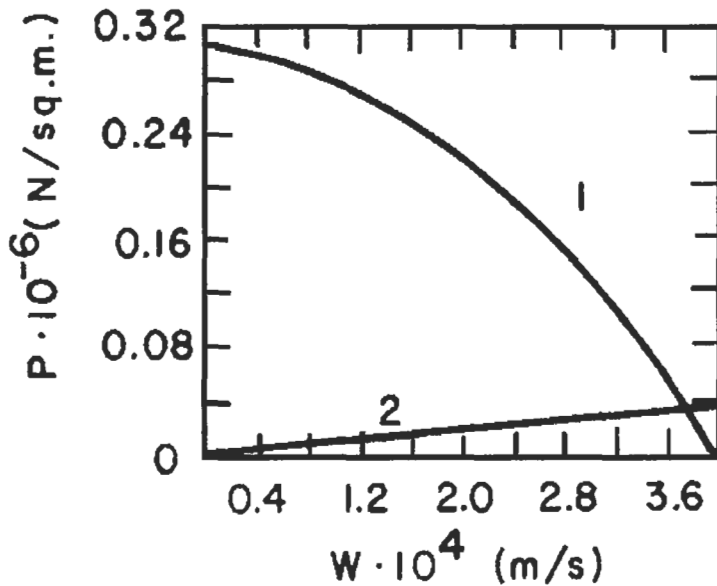
Figure 35. *Characteristic pump curve (1) and liquid static pressure on the boundary between the cake and filter plate (curve 2).*

Curve 2 in Figure 35 shows the relationship of liquid static pressure on the boundary between the cake and filter plate versus filtration rate. As seen from Figure 35, at any filtration rate, P_1 is spent for overcoming the resistance of filter plate P_{st}' and cake resistance. The intersection of curve 1 and line 2 corresponds to the start of filtration when there is no cake formed and the total pressure developed by the pump is spent for overcoming the resistance of the filter plate. It is assumed in this case that the filter plate resistance is independent of pressure. If it depends on pressure, the design should consider some average value, $P_{st}' = f(W)$, which may be determined from an experiment. For the limits from $P = 0$ to the increasing values $P = P_1 - P_{st}'$ (refer to Figure 34), curve 2 is obtained by means of graphical integration to show the dependence of the integral from forces compressing the cake. To determine q versus W at given y and x_w, we examine Figure 35 to find the values W_1P_1 and $P_1 - P_{st}'$; then for each value of $P - P_{st}' = P$ we determine from curve 2 the value of the integral. From this information, q can be determined. The results of these calculations are given in Figure 36. To determine $\tau = f(q)$, use Figure 36. This determines the value of the integral expression and, consequently, the value of τ. The data obtained in this manner are plotted in Figure 37. A more in-depth analysis of this case cane be found in *Fluid Mechanics and Unit Operations*, D. S. Azbel and N. P. Cheremisinoff, Ann Arbor Science Publishers, Mich. (1983). Additional references on the subject are also given in this reference.
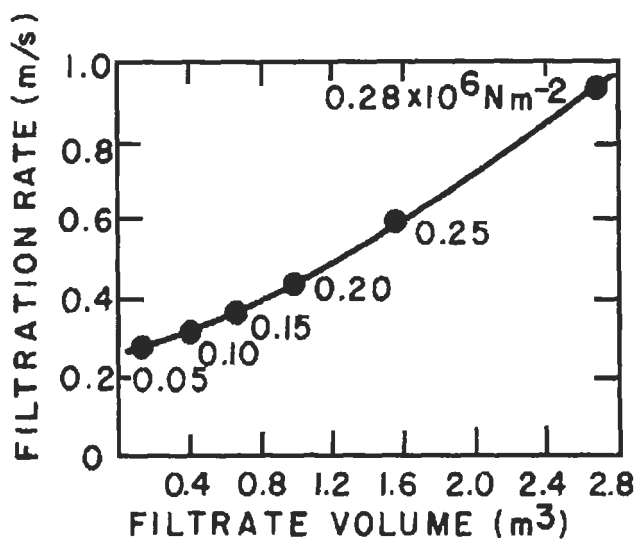
Figure 36. *Filtration rate vs. filtrate volume.*
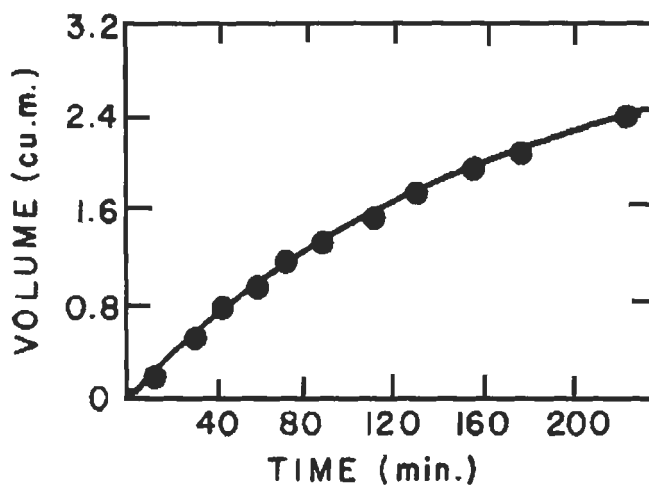
Figure 37. *Volume versus filtration time.*

The final method of operation to consider is that of filtration at constant pressure and constant rate. This method is employed when pure liquid is filtered through a cake of constant thickness and a constant pressure differential exists across the cake. Cake washing by displacement may be considered as filtering of a washing liquid through a constant cake thickness at constant pressure and flowrate. The rate of

washing is related to the rate of filtration during the final stages. Usually filtration during the final stages is performed under constant pressure. As such, expressions presented earlier are applicable. From filtration constants K and C, at constant pressure for a given system the filtration rate for the final period is determined. If the washing liquid passes through the filter following the same paths as the sludge and filtrate, then the difference between the washing rate and the filtration rate for this last period will mostly be due to a difference in viscosities of the wash liquor and the filtrate. Hence the expression $dV/d\tau = K/2(V + C)$ is applicable using the viscosity of the washing liquid, μ_w. Denoting the rate of filtration during the last period as $(dV/d\tau)_f$, then the washing rate cane be determined by:

$$(dV/d\tau)_w = (dV/d\tau)_f \, \mu/\mu_w$$

Designing for Optimum Filtration

The preceding discussions have focused on an analysis of the most common operating modes for solid-liquid filtration. The analysis however is general, and does not allow practical calculations without further development of working expressions and more careful consideration of proper boundary conditions and assumptions. We now focus on a series of special formulas developed specifically for assessing optimum conditions of filtration. These offer simplified calculation methods that can be readily performed on a PC, and in some cases with a hand-held calculator.

Optimization implies performing maximum filtration (removal efficiency and capacity) under the most economically favorable conditions (i.e, considering capital investment, O&M costs, labor costs, etc.). We will only provide formulas for filtration capacity. One of the tasks of the process engineer should be to develop sufficient vendor and operating data that will enable a careful cost analysis to be made for a filtration system. To increase filtration capacity, it is necessary to eliminate the cake from the filter plate as quickly as possible. However, this approach is only practical for continuous operations. In batch operations, the separation process must be interrupted to remove the cake. The operating cycle of a batch filter consists of a series of steps, namely, filter preparation, sludge loading, filtration, cake washing, drying and scraping. Filtration, washing and drying are basic operations whose duration time increases with filtrate volume and cake thickness. Filter preparation, sludge loading and removing are auxiliary operations, whose duration may be assumed to be independent of the filtrate volume and cake thickness. For any given filter, the duration of auxiliary operations is practically constant. Such an assumption can result in appreciable error in estimating the maximum filter capacity in some cases. To increase the capacity of batch filters

(with respect to the basic operations) it is sound practice to repeat the cycle as often as possible. This can be accomplished by feeding the filter with small portions of sludge. However, frequent filtration cycles mean an equal frequency of auxiliary operations, which is obviously time consuming and costly. Consequently, an optimum cycle duration that provides the maximum filter capacity must be determined for each application. In considering the general case for evaluating the maximum filter capacity at constant pressure, the operating cycle can be viewed as having four steps in series: filtration, washing, cake drying and removal. The filtration step has already been described by the formula:

$$V^2 + 2(R_f A/r_o x_o)V = (2\Delta P A^2/\mu\ r_o x_o)\tau$$

But the following empirical expression can be applied to describing washing:

$$G/G_o = 1 - 0.25/(V_{wf}/V_o)$$

where G = weight of dissolved substances in the washing liquid, G_o = weight of dissolved matter in filtrate retained in the cake before washing, V_{wf} = volume of washing liquid, and V_o = filtrate volume in the pores before washing.

The following equation describes the drying stage:

$$\tau_{dew}/C_{dew} = \{[(1 - m_o)^2 + 1]/2\} \times (m_e^{1-y})/(y - 1)$$

where τ_{dew} = dewatering time (s), C_{dew} = dewatering factor (s), m_o = residual cake saturation with liquid equal to the ratio of stationary liquid in the end of dewatering to the pore volume (fractions of unity), m_e = effective saturation with liquid equal to ratio of moving liquid volume to the total volume the same liquid and air (fractions of unity), and y = exponent, depending on the particle size in the cake. C_{dew} is the dewatering factor, that is defined as follows:

$$C_{dew} = \epsilon\mu r_o h_c^2/\Delta P$$

The dewatering factor, C_{dew}, combines the variables influencing the motion of liquids in the cake pores. The exponent y has been evaluated experimentally and literature-reported values range from 2.0 (for particles 1.8 mm in size) to 3.0 (for particles 0.09 mm in size). An average value of 2.5 may be assumed. A plot of

τ_{dew}/C_{dew} versus m_s for different values of m_o is shown in Figure 38. In this plot, τ_{dew}/C_{dew} is determined from the above equation at $y = 2.5$ and corresponding values of m_s (m_s = cake saturation with liquid, which is equal to the ratio of total stationary and moving liquid to pore volume, and is expressed in units of fraction of unity). This parameter can be calculated from the following equation:

$$m_s = (m_e - 2m_e m_o + m_o)/(1 - m_e m_o)$$

The curves shown permit evaluation of cake saturation by liquid rate versus dewatering time for a given set of conditions. During the dewatering stage, the volume of air sharply increases from zero to a maximum corresponding to the situation of air blowing through a dry cake. The amount of air passing through the pores as a function of dewatering time is given in Figure 39. In evaluating the optimum filtration time, we shall assume that the filter plate resistance is negligible. Furthermore, the duration of each stage will be evaluated as a function of filtrate volume on the assumption that all filtration conditions are constant. The time of filtration may be computed from earlier expressions.
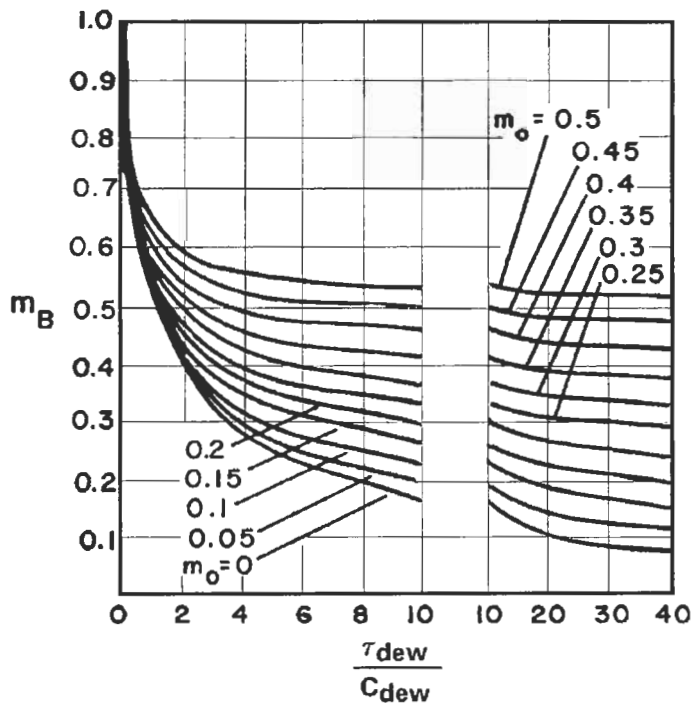
Figure 38. *Plot for determining cake saturation as a function of dewatering time.*
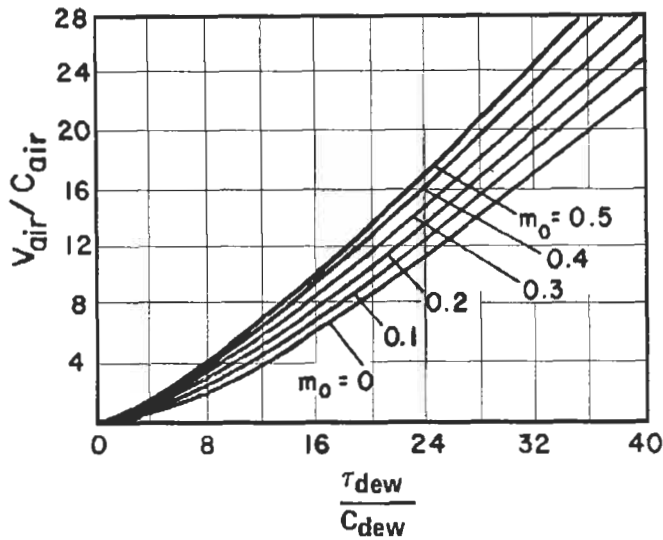
Figure 39. *Volume of blowing air vs. dewatering time.*

In the analysis we assume $R_f = 0$, and then $\tau = \frac{1}{2}(Bq^2)$, where $B = \mu r_o x_o / \Delta P$. The following relationships were developed and described by Azbel and Cheremisinoff (*Fluid Mechanics and Unit Operations*, Ann Arbor Science Publishers (1983)):

$$q_{wf} = 0.25 q_o / (1 - G/G_o)$$

As $q_o = \epsilon x_o q$, then $q_{wf} = D"q$, where:

$$D" = 0.25 \, \epsilon x_o / (1 - G/G_o)$$

Since the washing stage is essentially the case of filtration at constant cake thickness, q may be replaced by q_{wf}, and assuming the viscosity of the washing liquid to be the same as that of the filtrate, the following relationship between washing time and filtrate amount is developed:

$$\tau_w = BD"^2 q^2$$

Drying time as a function of filtrate amount may be estimated from:

$$\tau_{dew} = BC'q^2$$

where parameter C' is defined as:

$$C' = \tfrac{1}{2} \times \{(1 - m_o)^2 + 1\} \times \{(m_e^{1-y} - 1)/(y - 1)\} \times (\epsilon x_o)$$

The total time required for all three steps is:

$$\tau_b = \tau + \tau_w + \tau_{dew} = Eq^2$$

This may also be stated as:

$$q = (\tau_b/E)^{\frac{1}{2}}$$

where $E = B(\tfrac{1}{2} + D''^2 + C')$. The average filter capacity per unit time for a total filtration cycle may be characterized by a conditional average filtration rate obtained by dividing filtrate volume per cycle per unit filter area by the total cycle duration $(\tau_t = \tau_b + \tau_{aux})$:

$$u_{cond} = (\tau_b/E)^{\frac{1}{2}}/(\tau_b + \tau_{aux})$$

Assuming the filter plate resistance is negligible, the following expression applies for filtration without washing and drying steps:

$$\tau_b = \tau_{aux} + 2(\mu R_f^2 \tau_{aux}/2\Delta P r_o x_o)^{\frac{1}{2}}$$

This expression can be represented graphically in dimensionless form to simplify its use. It is generally expressed as the so-called "filtration number", defined as follows: $F_{fl} = \mu R_f^2 / 2\Delta P \tau_{aux} r_o x_o$. The filtration number, F_{fl}, is dimensionless and varies from zero at $R_f = 0$ to a large value when there is an increase in the viscosity of the sludge and R_f or a decrease in pressure drop, auxiliary time, specific cake resistance and the ratio of cake volume to filtrate volume. It may be assumed in practice that $F_{fl} = 0$ to 10. If washing and drying times are constant and independent of filtration time, they may be added directly to the auxiliary time. In

this case, the calculation of maximum filter capacity is similar to the case without washing and drying steps. From the times of the basic filtration steps, the filtrate volume per cycle can be computed along with the corresponding maximum capacity for different cases. The following equation can be used to determine the filtrate volume per cycle along with the corresponding maximum capacity for different trial cases in an evaluation:

$$q^2 + 2R_i q/r_o x_o = 2\Delta P\tau/\mu r_o x_o$$

For a cycle consisting of filtration and washing stages, and accounting for the filter plate resistance, the cake resistance corresponding to a maximum filter capacity is given by:

$$h_c = [2\Delta P x_o \tau/_{\mu ro}(1 + 2x_o L)]^{\frac{1}{2}}$$

where $L = \{\log(c_a/c_o)/K'_w\} \times \{\mu_{wf}/\mu\}$. In this parameter, c_a is the instantaneous concentration in washing liquid, and c_o is the concentration of solute in filtrate, K_w' is a constant which depends on the properties of the cake, the filtrate and the washing liquid, and hence must be determined from pilot tests. The following sample calculation summarizes the use of the major formulas.

Design Example for a Batch Nutsche Filter Operation: A batch Nutsche filter operates under constant pressure and maximum capacity with three stages: filtration, washing and drying. The Nutsche filter with a 1 m² filtration area operates under the following set of conditions:

- Viscosity of filtrate and washing liquid μ (N-s/m²) $= 10^{-3}$

- Air viscosity, μ_{air} (N-s/m²) $= 1.83 \times 10^{-5}$

- Surface tension of washing liquid, σ (N/m) $= 0.04$

- Cake specific resistance, r_o (m⁻²) $= 2 \times 10^{12}$

- Ratio of cake volume to filtrate volume, $x_o = 0.1$

- Pressure drop, ΔP (N/m²) $= 40,000$

- Porosity, $\epsilon = 0.45$

- Weight ratio G/G_o dissolved substance extracted by washing liquid to the substance in the cake before washing $= 0.98$

- Residual liquid saturation of cake m_o (approximate, considering the specific cake resistance) $= 0.5$

- Effective cake saturation with liquid at the end of drying, $m_e = 0.1$

- Exponent y in dewatering equations $= 2.5$

- Auxiliary time of operations, τ_{aux} (min.) $= 30$

- Maximum permissible cake thickness, h_c (m) $= 0.05$

The resistance of the filter plate is negligible. The problem is to determine the filtration, washing and drying times, the cake thickness, the volumes of filtrate, washing liquid and drying air. The solution to this design case is outlined below in steps.

1. The constant E is first determined:

$B = \mu r_o x_o / \Delta P = [10^{-3} \times 2 \times 10^{12} \times 0.1]/40,000 = 5 \times 10^3$

$D'' = 0.25 \, \epsilon x_o/(1 - G/G_o) = [0.25 \times 0.45 \times 0.1]/(1 - 0.98) = 0.56$

$C' = \frac{1}{2} \times \{(1 - m_o)^2 + 1\} \times \{(m_e^{1-y} - 1)/(y - 1)\} \times (\epsilon x_o) = 0.575$

$E = B(\frac{1}{2} \times D''^2 + C') = 5 \times 10^3(0.5 + (0.56)^2 + 0.575) = 6,945$

2. The filtrate volume is obtained, assuming $\tau_b = \tau_{aux}$, which corresponds to the maximum filter capacity: $q = (\tau_b/E)^{\frac{1}{2}} = (30 \times 60/6,945) = 0.51 \text{ m}^3$

3. Cake thickness: $h_c = x_o q = 0.10 \times 0.51 = 0.051 \text{ m}$. This corresponds to the maximum cake thickness.

4. Next determine the filtration time:

$\tau = 1/2Bq^2 = 0.5 \times 5 \times 10^3 (0.51)^2 = 650 \text{ s}$

5. The volume of washing liquid: $q_{wf} = D'' q = 0.56 \times 0.51 = 0.286 \text{ m}^3$

6. The washing time is: $\tau_w = B \times D''^2 \times q^2 = 5 \times 10^3 \times (0.56)^2 \times (0.51)^2 = 410 \text{ s}$.

7. Determine the drying time: $\tau_{dew} = B \times C' \times q^2 = 5 \times 10^3 \times 0.575 \times (0.51)^2 = 745 \text{ s}$. The total time of the filtration, washing and dewatering steps is approximately equal to the auxiliary time.

8. The amount of drying air is:

$C_{dew} = \epsilon \mu r_o h_c^2 / \Delta P = [0.45 \times 10^{-3} \times 2 \times 10^{12} \times (0.051)^2]/40,000 = 58.5 \text{ s}$. The ratio $\tau_{dew}/C_{dew} = 745/58.5 = 12.7$. From Figure 39, at mo $= 0.5$ and for $\tau_{dew}/C_{dew} = 12.7$, $V_a/C_a = 8.0$. The blowing factor is: $C_a = \epsilon h_c \mu/\mu_a = 0.45 \times 0.051 \times 10^{-3} \div 1.83 \times 10^{-5} = 1.25 \text{ m}^3/\text{m}^2$. Hence, the volume of blowing air is: Va $= 8 \times 1.25 = 10 \text{ m}^3/\text{m}^2$. Because ro $> 10^{11} \text{ m}^{-2}$, there is no need to introduce a correction factor for turbulence.

9. We next determine the residual saturation, m_o, by first calculating the dimensionless capillary number:

$K_p = \Delta P/r_o h_c \sigma = 40,000/(2 \times 10^{12} \times 0.51 \times 0.04) = 9.8 \times 10^{-6}$, where mo $= aK_p^{-0.264}$. Hence, $m_o = 0.025(9.8 \times 10^{-6})^{-0.254} = 0.55$. In this expression a $= 0.025$.
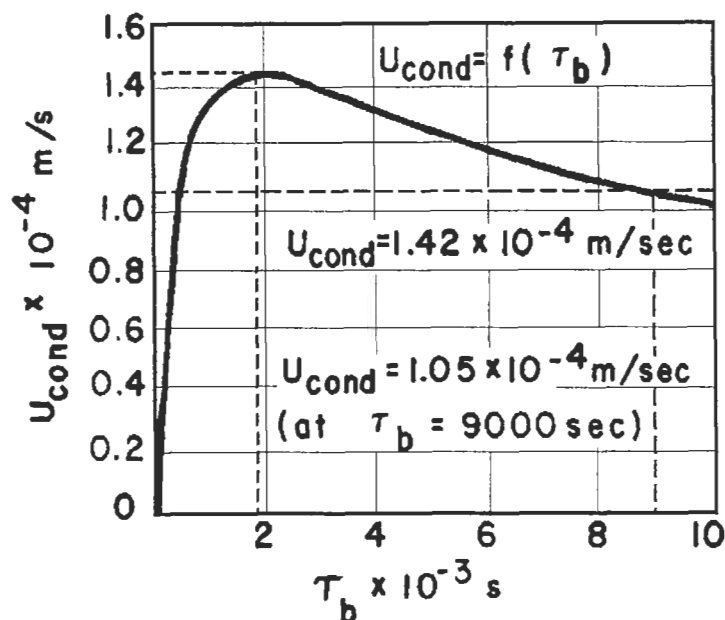
Figure 40. *Plot for Nutsche batch problem.*

Parameter a is a constant that takes into consideration end-effects as the filtrate flows through the system. It is important to evaluate the decrease in average filter capacity per unit time for the total operating cycle if the total time of filtration, washing and drying changes from zero to a value several times larger than the maximum filter capacity. We trace the curve obtained by a plot of τ_b versus u_{cond}. The plot is illustrated in Figure 40. This is a three-step process, namely filtration, washing and drying, under conditions of maximum capacity. The curve shows a shallow slope towards the right from a maximum. This means that even for a large increase in the basic steps, for $\tau_b = \tau_{aux}$, no significant decrease in filter capacity is observed. Thus, the increase in τ_b from 1,800 to 9,000 s results in a decrease in filter capacity from 1.42×10^{-4} to 1.05×10^{-4} m^3/m^2-s, or a 1.35 times reduction.

Example for Optimum Capacity of Batch Filers at Constant Rate: We now consider a filter whose operating cycle consists of a filtration step at constant rate and auxiliary steps for filter preparation, which includes sludge feeding and cake removal. The cake anf filter plate are assumed to be incompressible, which leads to two common cases. In the first case we consider a constant filtration process whose filtration time changes along with filtrate volume and cake thickness. In this situation, $\tau_b = q/u$, where u denotes a constant rate of filtration. The average filtration rate is defined as:

$$u_{cond} = q/(\tau_b + \tau_{aux})$$

And substituting for τ_b into the above, we obtain:

$$u_{cond} = 1/[1/u + \tau_{aux}/q]$$

For $q \to 0$ or $\tau_b \to 0$, $U_{cond} \to 0$, and at $q \to \infty$ or $\tau_b \to \infty$, $U_{cond} \to u$. That is, to increase the filter capacity, filtration time should be increased. However, such an increase is limited by the maximum allowable pressure drop which at constant operating conditions, establishes a maximum cake thickness. Let's consider the following example for the first case in this analysis. We wish to determine the capacity of a batch filter operating at a constant rate. The rate of filtration is q = 0. 1 X 10^{-3} m^3/m^2-s and the auxiliary time is 900 s. The solution to this problem is as follows. The filter capacity is characterized by the average conditional filtration rate, q_{cond}. Values of u_{cond} calculated are shown plotted in Figure 41. As shown, an increase in the amount of filtrate causes a sharp increase in filter capacity initially, with a limiting value of u attained eventually.

We now consider a second case, in which the filtration rate changes from one cycle to another; however, a constant rate is maintained during each cycle. The filtration is terminated when the pressure difference reaches a maximum allowable value. The amount of filtrate and cake thickness for each cycle will be different, as in Case 1, because the pressure difference depends not only on the cake thickness, but on the filtration rate as well. The following set of equations apply to this case:

$$u_{cond} = qu/(q + \tau_{aux}u)$$

$$q = \{\Delta P - \mu R_f u\}/\mu r_o x_o u$$
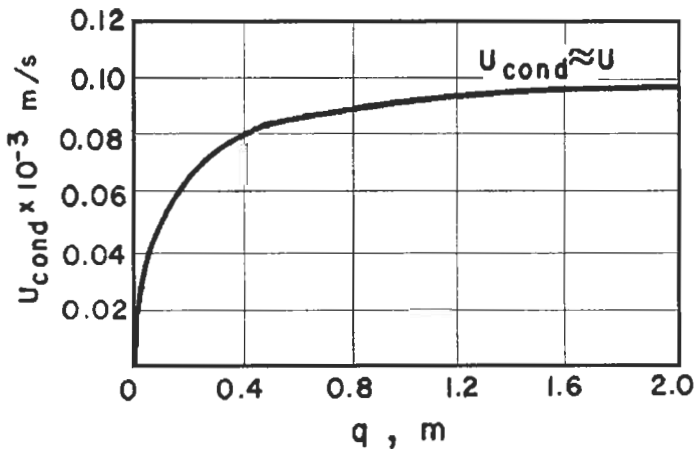
Figure 41. *Plot for example problem.*

In this model, ΔP is constant. This corresponds to the end of the filtration step. The following expressions apply:

$$u_{cond} = [\Delta Pu - \mu R_f u^2]/[\Delta P - \mu R_f u + \mu r_o x_o \tau_{aux} q^2]$$

$$u = \Delta P/[\mu R_f + (\mu r_o x_o \tau_{aux} \Delta P)^{\frac{1}{2}}]$$

The time of filtration corresponding to the maximum u_{cond} is:

$$\tau_b = \tau_{aux} + [(\mu R_f^2/\Delta P r_o x_o)\tau_{aux}]^{1/2}$$

To apply these equations, let's consider the following example. Determine a constant rate of filtration and the time of operation corresponding to the maximum capacity of a batch filter having the following conditions: maximum permissible pressure difference $\Delta P = 9 \times 10^4$ N/m^2; sludge viscosity $\mu = 10^{-3}$ N-s/m^2; filter plate resistance $R_f = 56 \times 10^{10}$ m^{-1}; specific cake resistance $r_o = 3 \times 10^{13}$ m^{-2}; $x_o = 0.333$; auxiliary time $\tau_{aux} = 600$ s; maximum permissible cake thickness $h_c = 0.025$ m. The solution is as follows:

The constant filtration rate is computed from the above formula:
$u = 9 \times 10^4/[10^{-3} \times 56 \times 10^{10} + \{10^{-3} \times 3 \times 10^{13} \times 0.333 \times 600 \times 9 \times 10^4\} = 0.0695 \times 10^{-3}$ m/s.

The filtration time is computed:
$\tau_b = 600 + [(10^{-3}(56 \times 10^{10})^2/9 \times 10^4 \times 3 \times 10^{13} \times 0.333) \times 600]^{1/2} = 1,056$ s.

The amount of filtrate is $q = \tau_b \times u = 0.0695 \times 10^{-3} \times 1,056 = 0.073$ m.

Finally, the cake thickness is: $h_c = q \times x_o = 0.073 \times 0.333 = 0.025$ m.

The reader will find additional design calculation examples in the two literature citations provided in this section. in addition, refer to the Suggested Readings section of this chapter for further information.

SEDIMENTATION EQUIPMENT

Thickeners and Clarifiers

There are a large number of processes in the chemical industries that handle a variety of suspensions of solid particles in liquids. The application of filtration techniques for the separation of these heterogeneous systems is sometimes very costly. If, however, the discrete phase of the suspension largely contains settleable particles, the separation can be effected by the operation of sedimentation. The process of sedimentation involves the removal of suspended solid particles from a liquid stream by gravitational settling. This unit operation is divided into *thickening,* i.e., increasing the concentration of the feed stream, and *clarification,* removal of solids from a relatively dilute stream.

A thickener is a sedimentation machine that operates according to the principle of gravity settling. Compared to other types of liquid/solid separation devices, a thickener's principal advantages include simplicity of design and economy of operation; its capacity to handle extremely large flow volumes; and versatility, as it can operate equally well as a concentrator or as a clarifier. In a batch-operating mode, a thickener normally consists of a standard vessel filled with a suspension. After settling, the clear liquid is decanted and the sediment removed periodically. The operation of a continuous thickener is also relatively simple. A drive mechanism powers a rotating rake mechanism. Feed enters the apparatus through a feed well designed to dissipate the velocity and stabilize the density currents of the incoming stream. Separation occurs when the heavy particles settle to the bottom of the tank. Some processes add flocculants to the feed stream to enhance particle agglomeration to promote faster or more effective settling. The clarified liquid overflows the tank and is sent to the next stage of a process. The underflow solids are withdrawn from an underflow cone by gravity discharge or pumping. A cross-sectional view of the rake arm of the thickener is illustrated in Figure 42. Thickeners can be operated in a countercurrent fashion. Applications are aimed at the recovery of soluble material from settleable solids by means of continuous countercurrent decantation (CCD). The basic scheme involves streams of liquid and thickened sludge moving countercurrently through a series of thickeners. The thickened stream of solids is depleted of soluble constituents as the solution becomes enriched. In each successive stage, a concentrated slurry is mixed with a solution containing fewer solubles than the liquor in the slurry and then is fed to the thickener. As the solids settle, they are removed and sent to the next stage. The overflow solution, which is richer in the soluble constituent, is sent to the preceding unit. Solids are charged to the system in the first-stage thickener, from which the final concentrated solution is withdrawn. Wash water or virgin solution is added to the last stage, and washed solids are removed in the underflow of this thickener.
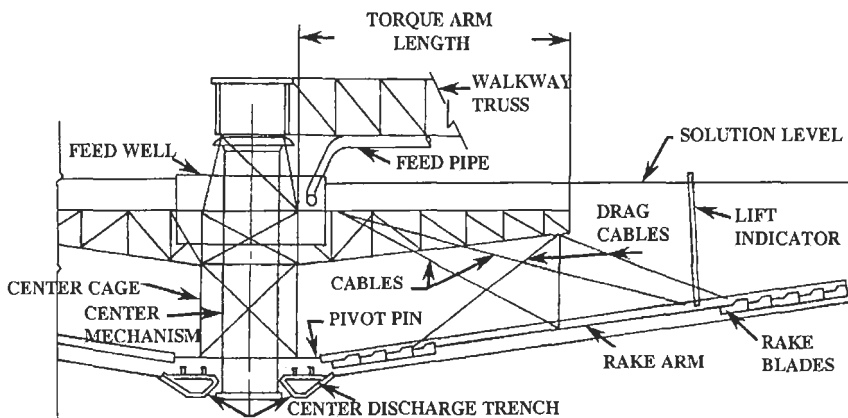
Figure 42. *Cross-section of a thickener.*
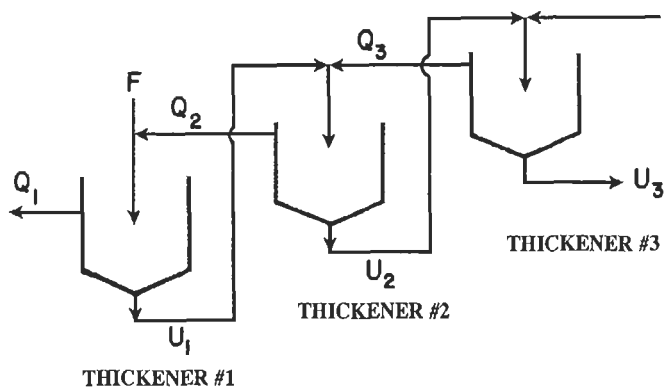
Figure 43. *Simplified flow scheme for a three-stage CCD system.*

The flow scheme for a three-stage CCD system is illustrated in Figure 43. The feed stream, F, is mixed with overflow Q2 (from thickener 2) before entering stage 1. The overflow of concentrated solution, Q1, is withdrawn from the first stage. The underflow from the first stage, U1, is mixed with third-stage overflow, Q3, and fed to the second stage. Similarly, the second-stage underflow, U2, is mixed with wash water and fed to thickener 3. The washed solids are removed from the third stage as the final underflow, U3.

Continuous clarifiers handle a variety of process wastes, domestic sewage and other dilute suspensions. They resemble thickeners in that they are sedimentation tanks or basins whose sludge removal is controlled by a mechanical sludge-raking mechanism. They differ from thickeners in that the amount of solids and weight of thickened sludge are considerably lower. Figure 44 shows one of the various types of cylindrical clarifiers. In this type of sedimentation machine, the feed enters up through the hollow central column or shaft, referred to as a siphon feed system. The feed enters the central feed well through slots or ports located near the top of the hollow shaft. Siphon feed arrangements greatly reduces the feed stream velocity as it enters the basin proper. This tends to minimize undesirable cross currents in the settling region of the vessel. Most cylindrical units are equipped with peripheral weirs; however, some designs include radial weirs to reduce the exit velocity and minimize weir loadings. The unit shown also is equipped with adjustable rotating overflow pipes.

Pre-settling operations are often performed in wastewater treatment applications. These can be as simple as rectangular settling basis, like the ones shown in the photograph of Figure 45. These basins serve to remove grit and other heavy particle prior to clarification. Chemical treatment may or may not be used at this stage of the water treatment operation. In general, this can be thought of as a pretreatment stage, prior to clarification and thickening operations.

Figure 44. *Clarifier in operation in a wastewater treatment plant at the Avtovaz auto-making plant in Russia.*

Figure 45. *Rectangular settling basins in operation.*

The total solids in municipal wastewaters exist in a distribution of sizes from individual ions up to visible particles. Specific analytical procedures have been established to distinguish the suspended fraction of the total solids and to further distinguish the settleable fraction within the suspended solids. A typical concentration of SS (suspended solids) for raw domestic wastewaters is 200 mg/l, but this can vary substantially from system to system. The lower limiting size for the SS fraction (about 1.5 microns) is arbitrarily defined by the test procedures and it should be noted that variations in test procedures themselves can also lead to

widely varying results, especially at the low solids levels characteristic of treated effluents. An analysis of a typical municipal wastewater in terms of solid distribution is given in Table 2.

Table 2. *Solids Distribution in Municipal Wastewaters.*

Fraction	Size Range, microns	Raw Wastewater		Secondary Effluent	
		Total Solids, mg/l	Volatile Matter, mg/l	Total Solids, mg/l	Volatile Matter, mg/l
Soluble	< 0.001	351	116	312	62
Colloidal	0.001 ~ 1	31	23	8	6
Supra-Colloidal	1 ~ 100	57	43	28	24
Settleable	> 100	74	59	0	0

The settleable and supracolloidal fractions together are essentially equivalent to the suspended fraction referred to above. Dividing lines between fractions again are somewhat arbitrary depending on tests applied, and overall concentrations in different fractions can vary substantially between systems depending on factors such as water use, travel time in sewers, ground-water infiltration, and prevalence of home garbage grinding. Contributions of dissolved, colloidal and suspended solids from individual homes, multi-family dwellings or other point sources often have concentrations two or more times the average for a whole system. In addition to particle size, specific gravity and strength or shear resistance of wastewater solids may affect solids separation performance.

The three basic types of solids separation processes are gravity separation, physical straining, and granular media filtration. Wastewater solids characteristics can be altered to enhance performance of the separation processes. The processes of chemical treatment (precipitation and/ or coagulation) and physical treatment (flocculation) are aimed at alteration of solids characteristics. In addition, during the separation processes themselves, agglomeration and compaction of solids generally continues, increasing separation efficiency and reducing the volume of separated solids.

Biological wastewater treatment processes also affect solids characteristics and hence solids separation. Activated sludge solids have been found to have a distinct bimodal distribution with one mode in the supracolloidal to settleable range and another near the border between the colloidal and supracolloidal fractions. The concentrations and size limits in each range are affected by conditions in the

biological reactor. Bacteria cellular debris, etc. fall into the finer (colloidal-supracolloidal) range. Agglomeration of these finer solids generally increases the efficiency of subsequent separation processes.

Processes for SS separation may fill three distinct functions in wastewater treatment, namely, pretreatment to protect subsequent processes and reduce their loadings to required levels, treatment to reduce effluent concentrations to required standards, and separation of solids to produce concentrated recycle streams required to maintain other processes. In the first two functions effluent quality is the prime consideration, but where the third function must be fulfilled along with one of the others, design attention must be given to conditions for both the separated solids (sludge) and the process effluent.

Wedge-wire screens can operate at very high hydraulic and solids loadings, but do not greatly reduce SS (suspended solids). Hence, wedge wire screens are limited to pretreatment applications where subsequent processes will assure production of a satisfactory final effluent. They can be considered as an adjunct to primary sedimentation or, where conditions prescribe, as an alternative. Sedimentation units must operate at relatively low hydraulic loadings (large space requirements), but can accept high solids loadings. With proper chemical or biological pretreatment and design, they can produce good quality effluents.

Microscreens and granular-media filters, operating at significantly higher hydraulic loads than sedimentation units, can produce an effluent with lower SS than is possible with sedimentation alone. In general they are not designed to accept high solids loadings, and are normally used following other processes which put out relatively low effluent SS concentrations. Selection of one of the alternative processes can be based on cost only where all factors not reflected in cost are equivalent. Direct cost comparison of individual solids removal processes usually proves impossible because of differences in factors such as: 1) effluent quality, 2) pretreatment requirements, 3) effects on sludge processing, 4) housing, space and head requirements. Meaningful cost comparisons usually involve practically the entire process configuration of the treatment facility, including processes for disposal of solid residues, and reflect how the individual unit processes affect one another. Some important cost considerations include: (1) Where chemical treatment is used to remove BOD or phosphates or improve SS removals, significant quantities of chemical sludge are produced. The cost of disposal of this sludge must be considered in process selection unless configurations being compared involve similar chemical treatment. The actual cost involved will depend greatly on the particular method of sludge disposal to be used; (2) Head Requirements - Some of the processes employed for SS separation (sedimentation, microscreens, etc.) require relatively small head (only 2 to 3 ft. to overcome losses at inlet and effluent controls and in connecting piping). Others, such as granular-media filters, and wedge-wire screens, require greater differential head (10 ft or more). Differences in head requirements are most significant where they necessitate capital outlay for an extra pumping step. The costs for pumping, however, even with lifts above 10

ft are usually not large in relation to the overall costs for treatment facilities; (3) Flow Variation - Both the rate and characteristics of the inflow to most treatment plants vary significantly with time. Diurnal cycles are found in all domestic discharges. Weekly and seasonal cycles are common in municipal systems as are variations between wet and dry weather. Even where only domestic flows are involved, the magnitude of variations can differ widely between different systems depending on system configuration, water use habits of the population and opportunities for groundwater infiltration or direct inflow of surface or subsurface drainage.

Industrial and institutional flows where significant, can further alter domestic patterns. Because of these wide differences, design of treatment facilities should be based, whenever possible, on measurements of actual flow variations in existing systems. Projected flow variations from existing systems should reflect elimination of excessive flows. Flows tend to be less variable in larger systems, due chiefly to differing times of travel from different sections and to damping effects of flow storage in large sewers. Equalization storage can be used to reduce diurnal variations in flow and in concentration of SS or other wastewater characteristics. Storage may also be used to handle peaks caused by direct inflow to the sewers during wet weather. Assuming that equivalent performance can be obtained either by increasing the size of treatment facilities or by providing equalizing basins, selection between these approaches can be based on their relative costs and environmental impacts. In plants using processes involving large, short-term recycle flows-such as for backwashing granular media filters-equalization is almost always justified.

Chemical Treatment and Settling Characteristics

As noted earlier, chemical coagulation and flocculation are common operations in wastewater treatment applications that are accomplished by a combination of physical and chemical processes which thoroughly mix the chemicals with the wastewater and promote the aggregation of wastewater solids into particles large enough to be separated by sedimentation, flotation, media filtration or straining. The strength of the aggregated particles determines their limiting size and their resistance to shear in subsequent processes. For particles in the colloidal and fine supra colloidal size ranges (< 1 to 2 microns) natural stabilizing forces (electrostatic repulsion, physical separation by absorbed water layers) predominate over the natural aggregating forces (van der Waals) and the natural mechanism (Brownian movement) which tends to cause particle contact. Coagulation of these fine particles involves both destabilization and physical processes which disperse coagulants and increase the opportunities for particle contact. Chemical coagulants used in wastewater treatment are generally the same as those used in potable water treatment and include: alum, ferric chloride, ferric sulfate, ferrous chloride, ferrous sulfate and lime. The effectiveness of a particular coagulant varies in different

applications, and in a given application each coagulant has both an optimum concentration and an optimum pH range. In addition to coagulants themselves, certain chemicals may be applied for pH or alkalinity adjustment (lime, soda ash) or as flocculating agents (organic polymers). For full effectiveness chemical coagulation requires initial rapid mixing to thoroughly disperse the applied chemicals so that they can react with suspended and colloidal solids uniformly.

Destabilization Mechanisms: The destabilizing action of chemical coagulants in wastewater may involve any of the following mechanisms: electrostatic charge reduction by adsorption of counter ions, inter-particle bridging by adsorption of specific chemical groups in polymer chains, and physical enmeshment of fine solids in gelatinous hydrolysis products of the coagulants.

Electrostatic Charge Reduction: Finely dispersed wastewater solids generally have a negative charge. Adsorption of cations from metal salt coagulants (in the case of iron and aluminum from their hydrolysis products), or from cationic polymers can reduce or reverse this charge. Where electrostatic charge reduction is a significant destabilization mechanism, care must be taken not to overdose with coagulant. This can cause complete charge reversal with restabilization of the oppositely charged coagulant-colloid complex.

Interparticle Bridging: When polymeric coagulants contain specific chemical groups which can interact with sites on the surfaces of colloid particles, the polymer may adsorb to and serve as a bridge between the particles. Coagulation using polyelectrolyte of the same charge as the colloids or non-ionic polymers depends on this mechanism. Restabilization may occur if excessive dosages of polymer are used. In this case all sites on the colloids may adsorb polymer molecules without any bridging. Excessive mixing ran also cause restabilization by fracture or displacement of polymer chains.

Enmeshment in Precipitated Hydrolysis Products: Hydroxides of iron, aluminum or, at high pH, magnesium form gelatinous hydrolysis products which are extremely effective in enmeshing fine particles of other material are formed by reaction of metal salt coagulants with hydroxyl ions from the natural alkalinity in the water or from added alkaline chemicals such as lime or soda ash. Sufficient natural magnesium is frequently present in wastewater so that effective coagulation is obtained merely by raising the pH with lime. Organic polymers do not form hydrolysis products of significance in this mechanism. At a pH value lower than that required to precipitate magnesium, the precipitates produced by lime treatment are frequently ineffective in enmeshing the colloidal matter in wastewater. The remedy for this condition generally involves the addition of low dosage of iron salts or polymers as coagulant aids both to destabilize and to increase the probability of enmeshment of colloids.

Use of Coagulants: Coagulants may also react with other constituents of the wastewater, particularly anions such as phosphate and sulfate, forming hydrolysis products containing various mixtures of ions. The chemistry of the reactions is extremely complex and highly dependent on pH and alkalinity. The presence of

high concentrations of these anions may require increased doses of coagulants or pH adjustment to achieve effective removals of suspended solids (SS). The design of chemical treatment facilities for SS removal must take into account: (1) the types and quantities of chemicals to be applied as coagulants, coagulant aids and for pH control and (2) the associated requirements for chemical handling and feeding, and for mixing and flocculation after chemical addition. The selection of coagulants should be based on jar testing of the actual wastewater to determine dosages and effectiveness, and on consideration of the cost and availability of different coagulants. Where expected changes in waste characteristics or market conditions may favor different coagulants at different times, chemical feed and handling should be set up to permit a switchover. In developing a testing program, general information on experience at other locations and on costs should be considered to aid in selection of processes and coagulants to be tested. Aluminum or iron salts tend to react with soluble phosphate preferentially so that substantial phosphorus removal must be involved before organic colloids can be destabilized. Required dosages will be affected by phosphorus content. Similarly, lime treatment to a pH at which coagulation is effective precipitates substantial phosphorus. Because chemical dosage and pH range for optimum SS removal may differ somewhat from those for optimum phosphorus removal, coagulant requirements may be determined by the effluent criteria for either pollutant, depending on wastewater characteristics and the choice of chemical.

Sludge Production: Chemical coagulation increases sludge production in sedimentation units due both to greater removal of influent suspended solids and to insoluble reaction products of the coagulation itself. The weight of sludge solids can be estimated by calculation of the sum of the expected SS removal and of the precipitation products expected from the coagulant dosages applied. Usually jar tests can be employed to obtain the necessary information for this calculation.

pH Control and Alkalinity: The critical factor in the control of lime reactions is pH. The pH for optimum effectiveness of lime coagulation, determined from jar testing and process operating experience can be used as a set point for a pH control of lime dosing. Alum and iron salt coagulation are much less sensitive to pH. Testing can determine optimum dosages for coagulation and whether natural alkalinity is adequate for the reactions. If supplemental alkalinity is needed either regularly or on an intermittent basis (e.g. during high wet weather flows) provisions should be included for feeding necessary amounts of lime or soda ash.

Points of Chemical Addition: In independent physical-chemical treatment or in phosphate removal in the primary clarifier ahead of biological treatment, chemicals are added to raw sewage. In tertiary treatment for phosphate removal and suspended solids (SS) reduction, they are added to secondary effluent. In both cases, proper mixing and flocculation units are needed. For phosphate removal or improvement of SS capture in biological secondary treatment, chemicals are often added directly to aeration units or prior to secondary settling units, without separate mixing and flocculation. In some phosphate removal applications coagulants are added at

multiple points, e.g. prior to primary settling and as part of a secondary or tertiary treatment step.

Supplementary Coagulants: Addition of the hydrolyzing metal coagulants to wastewater often results in a small slow-settling floc or precipitate of phosphorus. Additional treatment is required to produce a water with low residual suspended solids. Polymeric coagulants are beneficial in aggregating the precipitation products to a settleable size and increasing the shear strength of the floc against hydraulic breakup.

Coagulation Control: Because coagulation represents a group of complex reactions, laboratory experimentation is essential to establish and maintain the optimum coagulant dosage and to determine the effects of important variables on the quality of coagulation of the wastewater under investigation. With alum and iron coagulants two procedures are generally followed for this purpose: the jar test and measurement of zeta potential. Proper control of lime coagulation may be maintained by measuring the pH or automatically titrating alkalinity after lime addition.

The single, most widely used test to determine coagulant dosage and other parameters is the jar test, which attempts to simulate the full scale coagulation-flocculation process and has remained the most common control test in the laboratory since its introduction in 1918. Since the intent is to simulate an individual plant's conditions, it is not surprising that procedures may vary but generally have certain common elements. The jar test apparatus consists of a series of sample containers, usually six, the contents of which can be stirred by individual mechanically operated stirrers. Wastewater to be treated is placed in the containers and treatment chemicals are added while the contents are being stirred. The range of conditions, for example, coagulant dosages and pH, are selected to bracket the anticipated optima. After a 1 to 5 minute period of rapid stirring to ensure complete dispersion of coagulant, the stirring rate is decreased and flocculation is allowed to continue for a variable period, 10 to 20 minutes or more, depending on the simulation. The stirring is then stopped and the flocs are allowed to settle for a selected time. The supernatant is then analyzed for the desired parameters. With wastewater, the usual analyses are for turbidity or suspended solids, pH, residual phosphorus and residual coagulant. If desired, a number of supernatant samples may be taken at intervals during the settling period to permit construction of a set of settling curves which provide more information on the settling characteristics of the floc than a single sample taken after a fixed settling period. A dynamic settling test may also be used in which the paddles are operated at 2 to 5 rpm during the settling period. This type of operation more closely represents settling conditions in a large horizontal basin with continuous flow. A simple apparatus can be constructed from tubing, rubber stoppers and small aquarium valves to permit rapid sampling of the supernatant. The unit is placed next to the sample jars at the beginning of the settling period with the curved stainless steel tubes dipping into the jars. At desired intervals the vent valve is covered with a finger, permitting vacuum

to draw samples into the small sample bottles. The needle valves are adjusted so that supernatant is drawn into all the bottles at the same rate. When sufficient sample is obtained, the vent is uncovered and the bottles are replaced with empties. The maximum sampling rate is about once per minute. Figure 46 shows characteristic types of settling curves which may be obtained. Curve A indicates a coagulation which produced a uniformly fine floc so small that at the end of 1 to 2 minutes settling, the supernatant had a turbidity equal to that of the starting water due, in part, to the fine floc which resisted settling. Settling was slow and the final turbidity was not satisfactory. Curve B represents the most common type of settling rate obtained. During the first 5 minutes, the settling rate was practically a straight line on a semi-logarithmic plot. Settling was rapid and clarification was satisfactory. The coagulation represented by curve C shows that a mixture of large rapid settling floc and small, slow-settling particles was obtained. Settling was rapid for the first two minutes, but with little further clarification after that. High residual turbidity may also have resulted from incomplete coagulation. Curve D represents the ultimate in coagulation. Practically all of the floc particles were so large and dense that 97 percent settled within three minutes. Sedimentation was essentially complete within that time since only 0.5 percent additional floc settled in the next 27 minutes. Final clarity of the supernatant was entirely satisfactory. Measurement of turbidity provides the most rapid indication of the degree of solids removal obtained. The recommended procedure for turbidity measurement is by light scattering; however, other methods varying from simple visual evaluation to measurement of light transmitted on a laboratory spectrophotometer can be used for purposes of comparison.

SETTLING TIME (minutes)
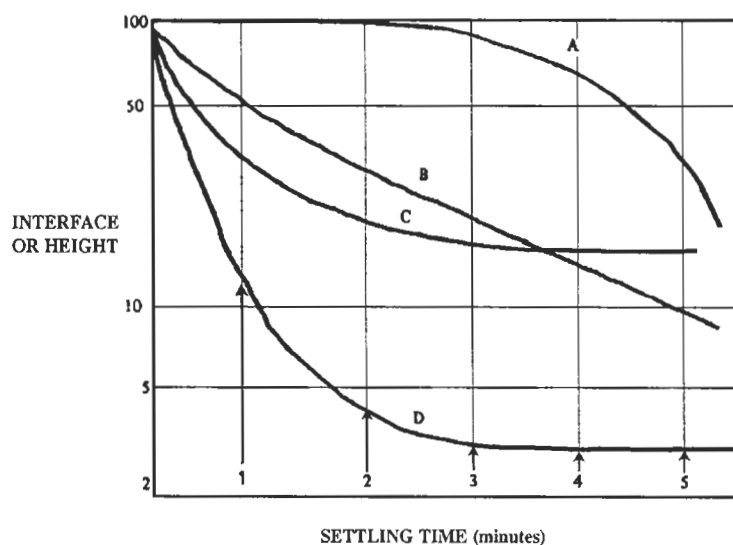
Figure 46. *Settling curves obtained from jar tests.*

Measurement of residual suspended solids is the only procedure which gives the actual weight concentration of solids remaining, but the procedure is too slow for purposes of process control. Where the character of the solids does not vary widely, their concentration generally correlates well with measured turbidity. A typical jar test might be run as follows: Wastewater samples are placed in containers and a rapid mix stage is started at 100 rpm. Selected dosages of coagulant covering the expected range of the optimum concentration are rapidly added to the containers and mixed for approximately 1 minute. If a polymer is to be used as a coagulant aid, it is usually added to each jar at or just before the end of the rapid mix. The paddles are then slowed to 30 rpm and mixing continues for 20 minutes. The paddles are then stopped and the sampling apparatus is placed in position. At settling times of 1, 3,5, 10 and possibly 20 minutes samples of supernatant are drawn for turbidity measurement. After the final turbidity sample is drawn, a larger volume of supernatant may be decanted for more complete analysis. If additional alkalinity is required to hold the coagulation in the optimum pH range, this should be added to the samples ahead of the coagulant unless automatic titrators are set up for pH control. Once an approximate optimum coagulant concentration has been determined, it may be desirable to repeat the jar test using that optimum with varying quantities of added alkalinity to give different pH values. Experience in coagulating a given wastewater provides the best guide as to methods for controlling the process.

Measurement of particle charge is another procedure which may be useful for control of the coagulation process. The total particle charge is distributed over two concentric layers of water surrounding the particle: an inner layer of water and ions which are tightly bound to the particles and move with them through the solution, and an outer layer which is a part of the bulk water phase and moves independently of the particles. Charges of these layers are not directly measurable, but the zeta potential, which is the residual charge at the interface between the layer of bound water and the mobile water phase, can be determined indirectly with commercially available instruments. In the zeta potential measurement procedure, a sample of treated water containing floc is placed in a special plastic cell under a microscope. Under the influence of a voltage applied to electrodes at the ends of the cell, the charged particles will migrate to the electrode having a polarity opposite that of the particle. The velocity of migration will be proportional to the particle charge and to the applied voltage. The particle velocity can be calculated by observing the time it takes a particle to travel a given distance across an ocular micrometer. The zeta potential can then be obtained from a chart which combines the particle velocity with instrumental parameters. Because of uncertainties in the constants relating charge and particle mobility, many test results are reported directly in terms of particle mobility. To control the coagulation by zeta potential, samples of water while being mixed are dosed with different concentrations of coagulant. Zeta potentials are then measured and recorded for floc in each sample. The dosage which produces the desired zeta potential value is applied to the treatment plant.

Zeta potentials of floc; produced in the plant may also be measured as a means of control. The zeta potential value for optimum coagulation must be determined for a given wastewater by actual correlation with jar tests or with plant performance. The control point is generally in the range of 0 to 10 millivolts. If good correlations can be obtained between some zeta potential values and optimum plant performance, then it is possible to make rapid measurements of particle charge to compensate for major variations in wastewater composition due to storm flows or other causes.

Sedimentation equipment is designed to perform two operations: to clarify the liquid overflow by removal of suspended solids and to thicken sludge or underflow by removal of liquid. It is the cross section of the apparatus that controls the time needed for settling a preselected size range of particles out of the liquid for a given liquid feed rate and solids loading. The area also establishes the clarification capacity. The depth of the thickener establishes the time allowed for sedimentation (i.e., the solid's residence time) for a given feed rate and is important in determining the thickening capacity. The clarification capacity is established by the settling velocity of the suspended solids.   Sedimentation tests are almost always recommended when scaling up for large settler capacities. By means of material balances, the total amount of fluid is equal to the sum of the fluid in the clear overflow plus the fluid in the compacted sludge removed from the bottom of the thickener. The average vertical velocity of fluid at any height through the thickener is the volumetric rate passing upward at that level divided by the unit's cross section. Note that if the particle settling velocity is less than the upward fluid velocity, particles will be entrained out in the overflow, resulting in poor clarification. For those size particles whose settling velocity approximately equals that of the upward fluid velocity, particles remain in a balanced suspension, i.e., they neither rise nor fall, and the concentration of solids in the clarification zone increases. This eventually results in a reduction of the settling velocity until the point where particles are entrained out in the overflow. The thickener must be designed so that the settling velocity of particles is significantly greater than the upward fluid velocity, to minimize any increase in the solids concentration in the clarification zone.

Solids concentration varies over the thickener's height, and at the lower levels where the solution is dense, settling becomes retarded. In this region the upward fluid velocity can exceed the particle settling velocity irrespective of whether this condition exists in the upper zone or not. Figure 47 illustrates this situation, where curve II denotes a higher feed rate. A proper design must therefore be based on an evaluation of the settling rates at different concentrations as compared to the vertical velocity of the fluid.

If the feed rate exceeds the maximum of the design, particulate matter are unable to settle out of the normal clarification zone. Hence, there is an increase in the solids concentration, resulting in hindered settling. The result is a corresponding decrease in the sedimentation rate below that observed for the feed slurry.
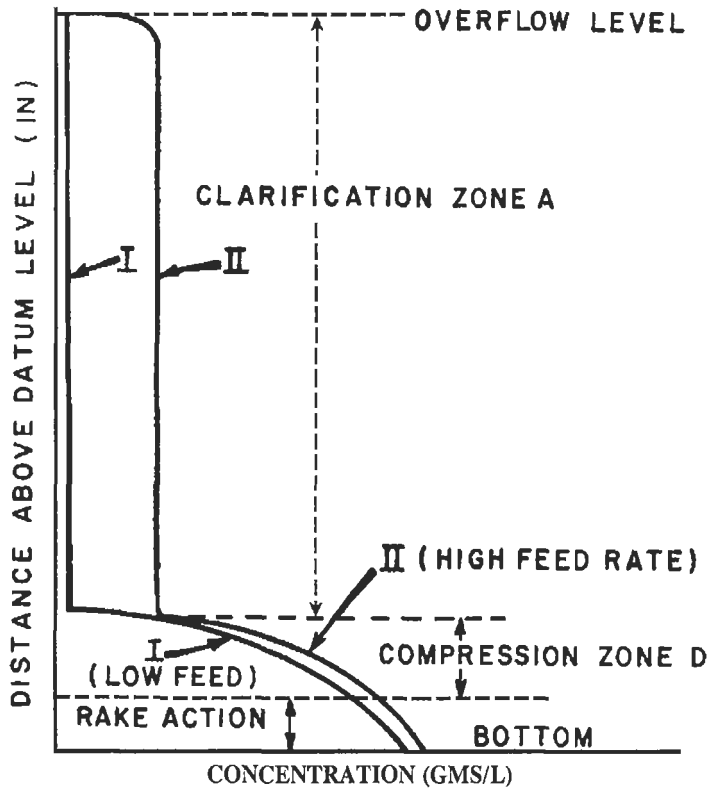
Figure 47. *Plot of concentration vs. height in a continuous sedimentation apparatus.*

The feed rate corresponding to the condition of just failing to initiate hindered settling represents the limiting clarification capacity of the system. That is, it is the maximum feed rate at which the suspended solids can attain the compression zone. The proper cross-sectional area can be estimated from calculations for different concentrations and checked by batch sedimentation tests on slurries of increasing concentrations. Figure 48 shows the effect of varying the underflow rate on the thickening capacity. In this example, the depth of the thickening zone (compression zone) increases as the underflow rate decreases; hence, the underflow solids concentration increases, based on a constant rate of feed. The curves of concentration as a function of depth in the compression zone are essentially vertical displacements of each other and are similar to those observed in batch sedimentation. When the sludge rakes operate, they essentially break up a semirigid structure of concentrated sludge. Generally, this action extends to several inches above the rakes and contributes to a more concentrated underflow.
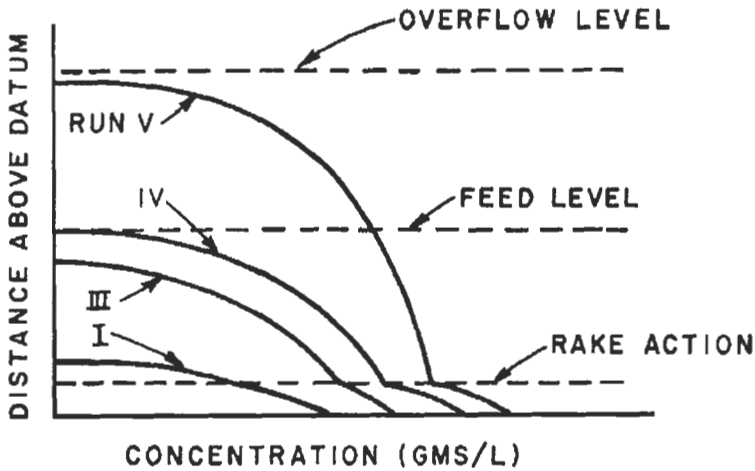
Figure 48. *Plot showing effect of underflow rate on thickening capacity.*

The required height of the compression zone may be estimated from experiments on batch sedimentation. The first batch test should be conducted with a slurry having an initial concentration equivalent to that of the top layer of the compression zone during the period of constant rate settling. This is referred to as the *critical concentration*. The time required for the sample slurry to pass from the critical concentration to the desired underflow concentration can be taken as the retention time for the solids in the continuous operation. The underlying assumption here is that the solids concentration at the bottom of the compression zone in the continuous thickener at any time is the same as the average concentration of the compression zone in the batch unit and at a time equal to the retention time of the solids in the continuous thickener. Hence, it is assumed that the concentration at the bottom of the thickener is an implicit function of the thickening time. The retention time is obtained from a batch test by observing the height of the compression zone as a function of time. The slope of the compression curve is described by the following expression:

$$- dZ/dt = k(Z - Z_\infty)$$

where Z, Z_∞ are the heights of compression at times t and infinity, respectively, and k is a constant that depends on the specific sedimentation system. Integrating this expression gives:

$$ln\ (Z - Z_\infty) = -kt + ln\ (Z_c - Z_\infty)$$

where Z_c is the height of the compression zone at its critical concentration. This equation is that of a straight line and normally is plotted as log $[(Z - Z_\infty)/(Z_0 - Z_\infty)]$ versus time, where Z_0 is the initial slurry concentration.

If batch tests are performed with an initial slurry concentration below that of the critical, the average concentration of the compression zone will exceed the critical value because it will consist of sludge layers compressed over varying time lengths. A method for estimating the required time to pass from the critical solids content to any specified underflow concentration is as follows: First, extrapolate the compression curve to the critical point or zero time. Then locate the time when the upper interface (between the supernatant liquid and slurry) is at height Z'_0, halfway between the initial height, Z_0, and the extrapolated zero-time compression zone height, Z'_0. This time represents the period in which all the solids are at the critical dilution and go into compression. The retention time is computed as $t - t_c$, where t is the time when the solids reach the specified underflow concentration. The procedure is illustrated in Figure 49. The determination of the required volume for the compression zone should be based on estimates of the time each layer has been in compression. The volume for the compression zone is the sum of the volume occupied by the solids plus the volume of the entrapped fluid. This may be expressed as:

$$V = Q(\Delta t)/\rho_s + \int\{m_t Q m_s \rho_i\}dt$$
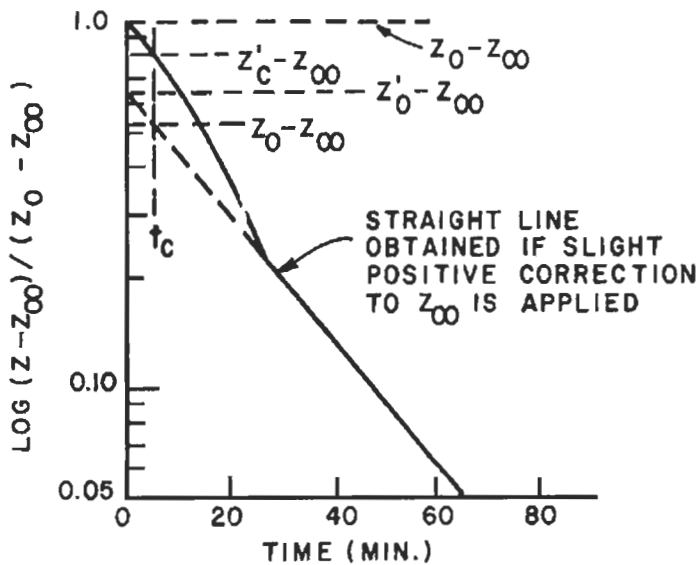
Figure 49. *Extrapolation of sedimentation data to estimate time for critical concentration.*

where: Q = solids mass feed per unit time
$\Delta t = t - t_c$ = retention time
m_ℓ = mass of liquid in the compression zone
m_s = mass of solids in the compression zone

An approximate solution to this expression can be obtained if we assume m_ℓ/m_s to be constant, i.e., an average mass ratio in the thickening zone from top to bottom. This results in the following expression:

$$V = Q\Delta t[1/_{\rho s} + (1/\rho_\ell)(m_\ell/m_s)_{avg}]$$

Particle Settling from Gas Suspensions

Settling equipment for removing solid and/or liquid particles from gas streams is designed for several reasons: cleaning of ventilation air or fly ash removal from flue gases; product-quality improvement; recovery of valuable products; powdered-product collection. The forces utilized for separating particles from gas streams may be classified as gravity settling, inertial deposition, flow-line interception, diffusional deposition, electrostatic deposition, thermal precipitation, and sonic agglomeration. We shall discuss gravitational deposition in the remainder of this section. Gravitational separation of particles from a gas suspension is achieved in a chamber in which the velocity of a gaseous suspension is reduced to enable particles (solid or liquid) to settle out by the action of gravity. To decrease the trajectory path of a particle and, consequently, the time of settling, horizontal plates are positioned within the chamber. These plates significantly improve the collection efficiency of the discrete phase. To establish the length of the chamber required to remove particles of a certain minimum size, or to determine the size particles removed by an existing chamber with a specified loading, the particle settling velocity must be evaluated. If the particulates in question follow Stokes' law and are approximately spherical, the familiar settling velocity expression may be applied:

$$u = d^2(\gamma_p - \gamma)/18\mu$$

where u is the particle settling velocity, μ is the gas viscosity, d is the mean particle diameter, and γ_p, γ are the particle and gas specific gravities, respectively. Since $\gamma_p \gg \gamma$, then $u \approx d^2\gamma_p/18\mu$. For very large particles (typically above several hundred microns), the so-called Allen equation is accurate in predicting settling velocity:

$$u = 0.153d^{1.14} \gamma_p^{0.71}/[(\gamma/g)^{0.29}\mu^{0.43}]$$

If, for a given heterogeneous system, the settling velocity of the smallest particles that will separate out is u and the height of the chamber (or the distance between horizontal plates) is H, then the settling time will be H/u. The time required for a

unit volume of gas to remain in the chamber, i.e., the gas residence time, must at least be equal to the settling time to allow the smallest particles to be removed. The linear gas velocity is equal to the volumetric flowrate, q, divided by the cross-sectional area of the chamber, F. For a chamber of length L, the residence time of the gas in the chamber is L(q/F). As this time must be equal to that of the smallest particle, we may equate the two:

$$L = Hq/uF$$

This equation shows that at a constant gas velocity, q/F, the length of the chamber, L, is proportional to the height of the chamber, H. Obviously, the smaller the height, the smaller the chamber length required for a desired separation, which explains the advantage of a multiplate design over a simple chamber. Because the smallest particles will settle according to Stokes' law, we may combine the above expressions:

$$L = 18Hq\mu/Fd^2\gamma_p$$

Thus, the length of a settling chamber is inversely proportional to the square of the particle diameter. For example, if it is desirable to separate out particles that are two times smaller than the selected size, then the length of the chamber must be increased by a factor of four. The equation may also be used to determine the smallest particle diameter that can be removed by a chamber of specified dimensions. The following example problem illustrates some of these design principles.

Example: Determine   the dimensions of a simple settling chamber required to remove 50 μ size particles under the following conditions: Gas capacity, q = 2400 m^3/hr; Particle density, ρ_p = 2400 kg/m^3; Gas temperature, t = 20 °C; Gas density, ρ = 1.2 kg/m^3; Gas viscosity, μ = 1.8×10^{-5} N-s/m^2 . The solution is as follows. The settling regime for the particles must be determined first. Hence, the critical particle diameter is computed first:
d_{cr} = 2.62[μ^2/(g(ρ_p - ρ)ρ)]$^{1/3}$ = 2.62×[(1.8×10^{-5})^2 ÷ (9.81×(2400 - 1.2)×1.2)]$^{1/3}$ = 5.9×10^{-5} m = 59 μ.
Since the size range of the particles to be removed is less than the critical diameter, we are confident that the particles will follow Stokes' law. Hence, the settling velocity for a 50 μ size particle is:
u = d^2(ρ_p - ρ)g/18μ = (50×10^{-6})^2×2400×9.81/(18×1.8×10^{-5}) = 0.182 m/s.
Assuming a rectangular chamber, the time for settling is: t = H/u. Denoting the chamber volume as V$_c$, then the residence time is: t = 3600V$_c$/q = 3600V$_c$/2400 = 1.5Vc. Hence, the maximum chamber height is: H = 1.5uV$_c$ = 1.5×0.182×V$_c$ = 0.273 V$_c$, m. The longitudinal cross section of the chamber is: F = V$_c$/H = V$_c$/2.73 V$_c$ = 3.7 m^2. To determine the specific dimensions of the chamber, the chamber's cross section must be assessed. This area is a function of the allowable

gas velocity through the chamber. Assuming a gas velocity, w, of 0.2 m/s, then the cross sectional area is $S = q/3600w = 2400/(3600 \times 0.2) = 3.33 \text{ m}^2$. As the two cross sections of the chamber are now known, by selecting one dimension, based on acceptable space requirements, the other dimensions can be established. For example, let $H = 1$ m, then $b = S/h = 3.33/1 = 3.33$ m, and $L = F/b = 3.7/3.33 = 1.11$ m.

CENTRIFUGAL SEPARATION EQUIPMENT

A very important group of separation techniques that rely on mechanical forces are those based on the application of centrifugal force to separate phases. The influence of a centrifugal field on particles in a suspension or on two liquids of widely different densities, is analogous to the separation achieved in the presence of a gravitational field. However, unlike gravity, which is essentially constant, the strength of a centrifugal field can be varied through changes in rotational speed or in equipment dimensions. The primary equipment used to perform this unit operation includes cyclones separators for gas suspensions, hydroclones for liquid-solid suspensions, and centrifuges for liquid-solid, liquid-liquid and gas-gas separations. In cyclone separators and hydroclones, the heterogeneous suspension is subjected to centrifugal force brought about by its own rotation with respects to a stationary apparatus boundary by introducing it through a tangential inlet. In centriguges, the suspension undergoes rotation with respect to a revolving apparatus boundary.

Cyclone Separators and Hydroclones

Among industry jargon, the terms cyclone and hydroclone are used interchangeably. In the strict sense, however, a cyclone is restricted to gas - particulate separations, whereas the hydroclone has been more traditionally applied to solid-liquid or slurry separations. Nonetheless, the design configurations, operating principles, and selection and sizing criteria are nearly identical. Both devices are the simplest and most economical separators (also called solids collectors or, simply - collectors). Their operations are identical, in which forces both of inertia and gravitation are capitalized on, and their primary advantages are high collection efficiency in certain applications, adaptability and economy in power. The main disadvantage lies in their limitation to high collection efficiency of large-sized particles only. In general, cyclones are not capable of high efficiencies when handling gas streams containing large concentrations of particulate matter less than 10 μ in size. Cyclones generally are efficient handling devices for a wide range of particulate sizes. They can collect particles ranging in size from 10 to above 2,000 μ with varying degrees of removal efficiency, with inlet loadings ranging from less than 1 gr/scfm to greater than 100 gr/scfm. There are many

design variations of the basic cyclone configuration. Because of the cyclone's/hydroclone's simplicity and lack of moving parts, a wide variety of construction materials can be used to cover relatively high operating temperatures of up to 2,000 °F. Cyclones are employed in the following general applications: collecting coarse dust particles; handling high solids concentration gas streams between reactors such as Flexicokers (typically above 3 gr/scf); for classifying particulate sizes; in operations in which extremely high collection efficiency is not critical; and as precleaning devices in line with high-efficiency collectors for fine particles. Because of the similarities between cyclones and hydroclones, the discussions for these equipment are not separated. Figure 50 shows a cutaway view of a typical industrial cyclone.
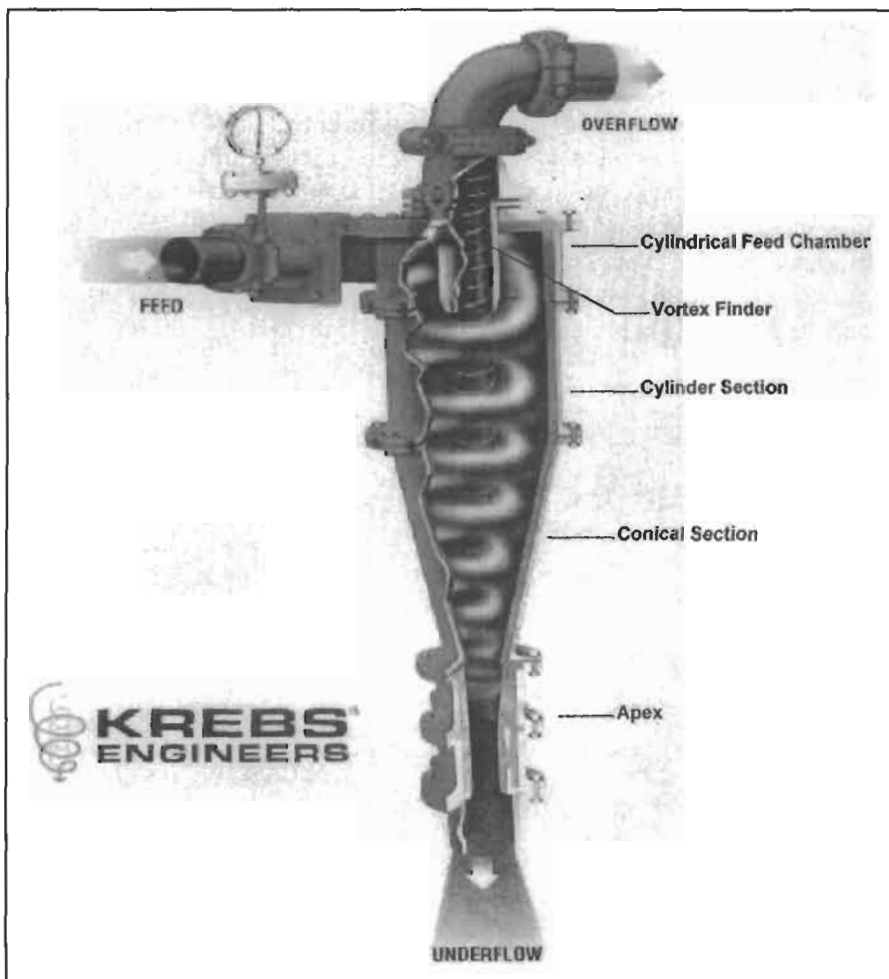
Figure 50. *Cutaway view of a cyclone separator. (Courtesy of Krebs Engineers, Menlo Park, CA).*

For many years cyclones have been extensively utilized in the classification of particles in comminution circuits. The practical range of classification for cyclones is 40 microns to 400 microns, with some remote applications as fine as 5 microns or as coarse as 1,000 microns. Cyclones are used in both primary and secondary grinding circuits as well as regrind circuits. The following discussions provide a means of selecting the proper number and size of cyclones and to determine the proper level of operating variables. Generally, it is recommended that cyclone suppliers be consulted for sizing confirmation. Some cyclone suppliers employ digital computers to aid in the sizing and selection of cyclones. Following Figure 50, during operation, the feed slurry or suspension enters the cyclone or hydroclone under pressure through the feed pipe into the top of the cylindrical feed chamber. This tangential entrance is accomplished by two types of design, as shown in Figure 51. Since the majority of research has been done with the involuted type, the graphs and relationships shown may not be strictly applicable to other designs. As the feed enters the chamber, a rotation of the slurry inside of the cyclone begins, causing centrifugal forces to accelerate the movement of the particles towards the outer wall. The particles migrate downward in a spiral pattern through the cylindrical section and into the conical section. At this point the smaller mass particles migrate toward the center and spiral upward and out through the vortex finder, discharging through the overflow pipe. This product, which contains the finer particles and the majority of the water (in the case of a hydroclone), is termed the overflow and should be discharged at or near atmospheric pressure.

TANGENTIAL FEED
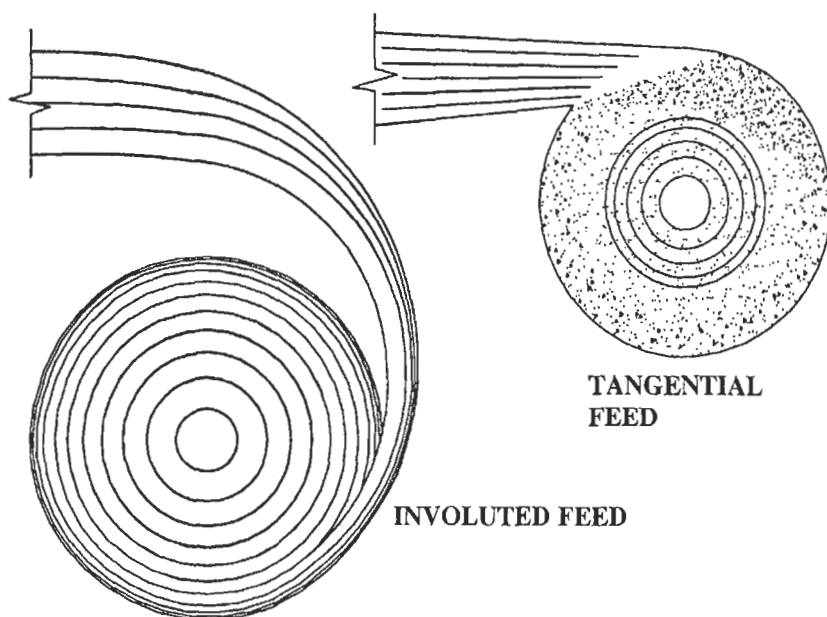
INVOLUTED FEED

Figure 51. *Involuted vs. tangential feeds.*

The higher mass particles remain in a downward spiral path along the walls of the conical section and gradually exit through the apex orifice. This product is termed the underflow and also should be discharged at or near atmospheric pressure. The definition of a "standard cyclone" is that cyclone which has the proper geometrical relationship between the cyclone diameter, inlet area, vortex finder, apex orifice, and sufficient length providing retention time to properly classify particles. As with the involuted type design, the graphs and mathematical relationships shown for proper selection and sizing of cyclones apply to the "standard cyclone" geometry. The main parameter is the cyclone diameter. This is the inside diameter of the cylindrical feed chamber. The next parameter is the area of the inlet nozzle at the point of entry into the feed chamber. This is normally a rectangular orifice, with the larger dimension parallel to the cyclone axis. The basic area of the inlet nozzle approximates 0.05 times the cyclone diameter squared. The next important parameter is the vortex finder. The primary function of the vortex finder is to control both the separation and the flow leaving the cyclone. Also, the vortex finder is sufficiently extended below the feed entrance to prevent short circuiting of material directly into the overflow. The size of the vortex finder equals 0.35 times the cyclone diameter. The cylindrical section is the next basic part of the cyclone and is located between the feed chamber and the conical section. It is the same diameter as the feed chamber and its function is to lengthen the cyclone and increase the retention time. For the basic cyclone, its length should be 100% of the cyclone diameter. The next section is the conical section, typically referred to as the cone section. The included angle of the cone section is normally between 10° and 20° and, similar to the cylinder section, provides retention time.

The termination of the cone section is the apex orifice. The critical dimension is the inside diameter at the discharge point. The size of this orifice is determined by the application involved and must be large enough to permit the solids that have been classified to underflow to exit the cyclone without plugging. The normal minimum orifice size would be 10% of the cyclone diameter and can be as large as 35%. Below the apex is normally a splash skirt to help contain the underflow slurry in the case of a hydroclone.

In determining the proper size and number of cyclones required for a given application, two main objectives must be considered. The first is the classification or separation that is required, and the second is the volume of feed slurry to be handled. In the case of hydroclones, before determining whether these objectives can be achieved, it is necessary to establish a base condition as follows: Feed liquid - water at 20 °C. Feed solids - spherical particles of 2.65 specific gravity; Feed concentration - less than 1 % solids by volume; Pressure drop - 69 kPa (10 psi); Cyclone geometry - "standard cyclone" as described above.

By convention, classification has been defined as the particle size of which 1 % to 3% reports to the cyclone overflow with coarser particles reporting to the cyclone underflow. Recent investigations reported by Arterburn (1999) have defined classification as the particle size of which 50% reports to the overflow and 50% to

classification as the particle size of which 50% reports to the overflow and 50% to the underflow, or the so-called D_{50c} point. Following Arterburn, Figure 52 shows the typical relationship between particle diameter and the percent recovered to underflow. The portion of the curve near the 50% recovery level is quite steep and lends itself readily to determining an accurate particle diameter. Examination of the recovery curve near the 97% to 99% recovery level shows that the curve is nearly horizontal and a small differential could change the micron diameter considerably. Note that the particle size shown on Figure 52 and also used for calculations in this example is defined as the minimum particle diameter of a given size band. For example, a particle that passes a 150 mesh screen (105 microns) but is retained on a 200 mesh screen (74 microns) would actually have a diameter between 74 microns and 105 microns. In this example, the size of 74 microns would be used for particles in this size range.
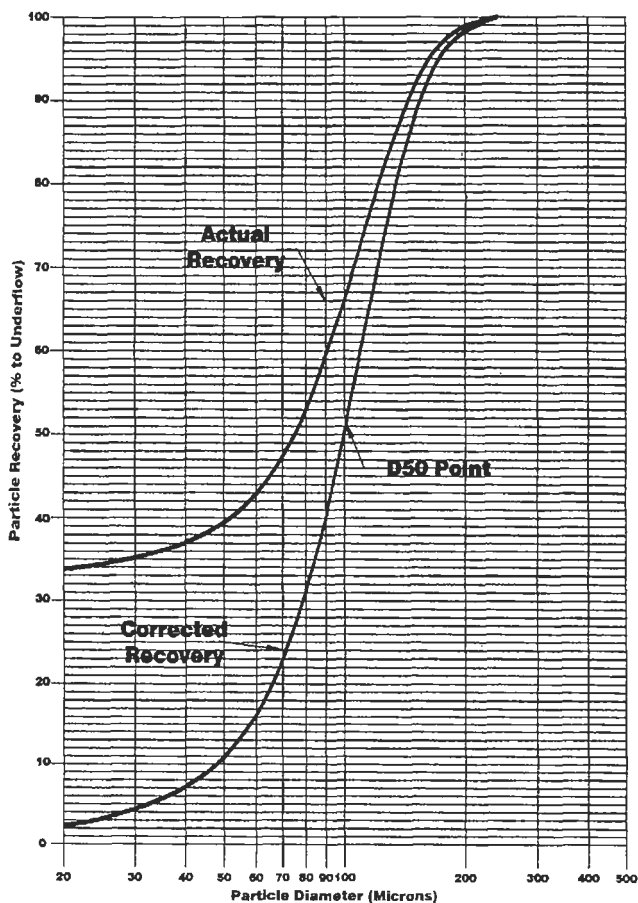
Figure 52. *Plot reported by Arterburn on particle diameter vs. particle recovery for hydroclone applications.*
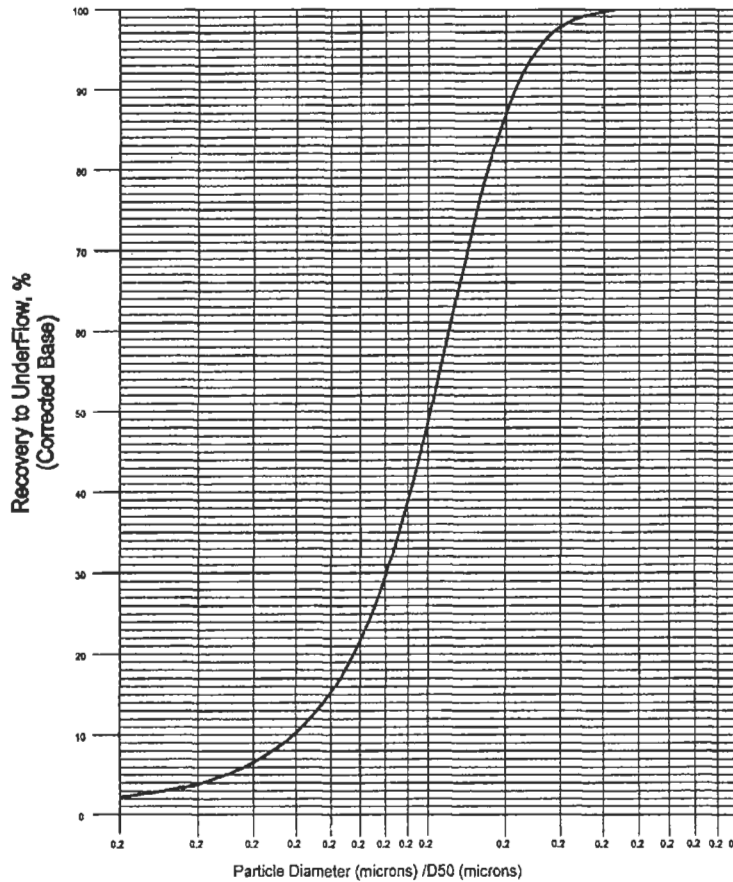
Figure 53. *Reduced particle recovery curve.*

Figure 52 also shows that the actual recovery curve does not decrease below a certain level. This indicates that a certain amount of material is always recovered to the underflow and bypasses classification. If a comparison is made between the minimum recovery level of solids to the liquid that is recovered, they are found to be equal. Therefore it is assumed that a percent of all size fractions reports directly to the underflow as bypassed solids in equal proportion to the liquid split. Then each size fraction of the actual recovery curve is adjusted by an amount equal to the liquid recovery to produce the "corrected recovery" curve shown in Figure 52. As the D_{50c} point changes from one application to another, the recovery curves shift, along the horizontal axis. In order to determine a single graph which represents the corrected recovery curve, the particle size of each size fraction is divided by the D_{50c} value and a "reduced recovery" curve can be plotted, as shown in Figure 53. Studies reported by Arterburn have shown that this curve remains constant over a wide range of cyclone diameters and operating conditions when applied to a slurry

containing solids of a single specific gravity and a typical or normal size distribution such as those encountered in most grinding circuits. The following equation gives a mathematical relationship which can be used to calculate the reduced recovery. This recovery, along with the bypassed solids, is used to predict the complete size distribution for the underflow product.

$$R_r = [e^{4d} - 1]/[e^{4d} + e^4 - 2]$$

where: R_r = Recovery to underflow on corrected basis
        d = Particle diameter /D_{50c} particle diameter

In designing comminution circuits the objective is to produce an overflow from the cyclone which has a certain size distribution, normally defined as a given percent passing a specified micron size. An empirical relationship shown in Table 3 can be used to relate the overflow size distribution to the D_{50c} required to produce the specified separation. The relationship of this table is for typical or average grinding size distributions and may vary slightly depending upon the grinding properties of a material such as ore.

Table 3. *Relationship of D_{50c} to Overflow Size Distribution.*

Required Overflow Size Distribution[a]	Multiplier[b]	Required Overflow Size Distribution[a]	Multiplier[b]
98.8	0.54	70.0	1.67
95.0	0.73	60.0	2.08
90.0	0.91	50.0	2.78
80.0	1.25		

(a) - Percent passing of specified micron size; (b) - To be multiplied times micron (μ) size.

As an example - to produce an overflow of 80 % passing 149 μ (100 mesh), the multiplier from Table 3 at 80 % passing is 1.25. The micron size for the application is 149 μ (100 mesh). The D_{50c} required = $1.25 \times 149 = 186$ μ for the application. The separation that a cyclone/hydroclone can achieve can be approximated from the following relation. The D_{C50} (base) for a given diameter cyclone is multiplied times a series of correction factors designated by C_1, C_2, and C_3:

$$D_{50c}(\text{application}) = D_{50c}(\text{base}) \times C_1 \times C_2 \times C_3$$

D_{50c} (base) is the micron size that a "standard cyclone" can achieve operating under the base conditions. This is given in Figure 54, as computed from the equation below. For example, a 25.4 cm (10 in.) Diameter cyclone has a bas D_{50c} point of

24 μ. In the formula below, D is the cyclone diameter in cm. The first correction C_1 defines the influence of the concentration of the solids contained in the feed slurry. The graphical representation of this correction is given in Figure 55.

$$D_{50c}(\text{base}) = 2.84 \times D^{0.66}$$

Figure 55 indicates that the level of percent solids is extremely important in determining the proper separation. the higher the concentration, the coarser the separation. The correction factor is a relative measure of slurry viscosity and is affected by such parameters as particle present and particle shape. A feed that contains a large amount of clay would tend to shift the curve to the left, resulting in a coarser separation. In contrast, the absence of fines would shift the curve to the right and result in a finer separation.
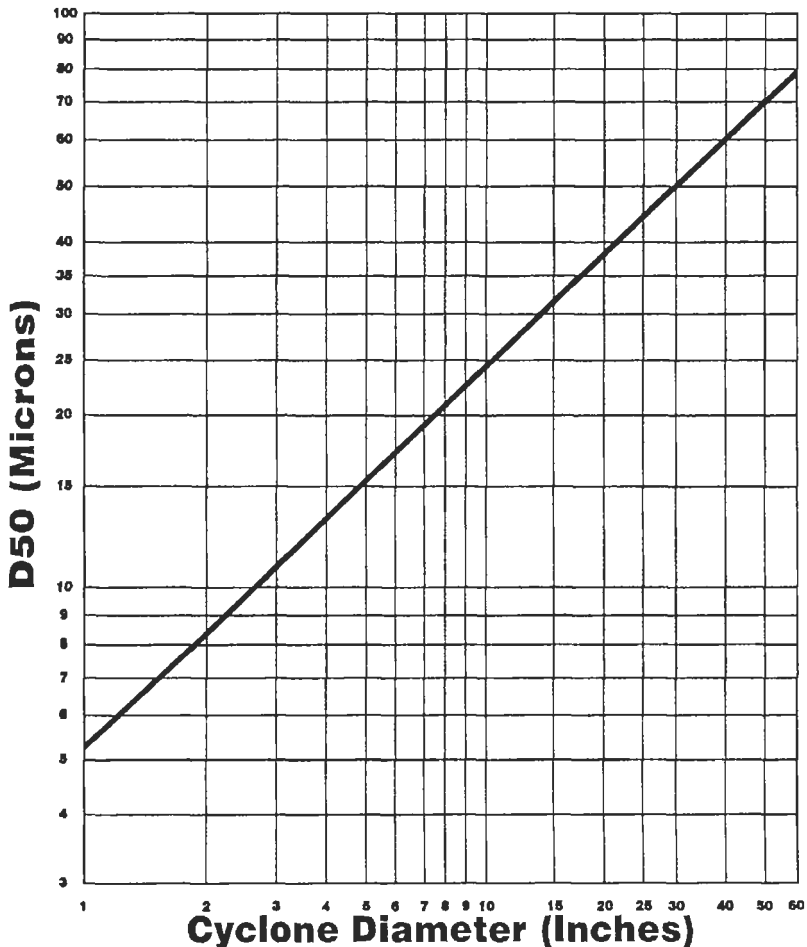
Figure 54. *Cyclone diameter vs.* D_{50c} *for "typical cyclones" - after Arterbern.*
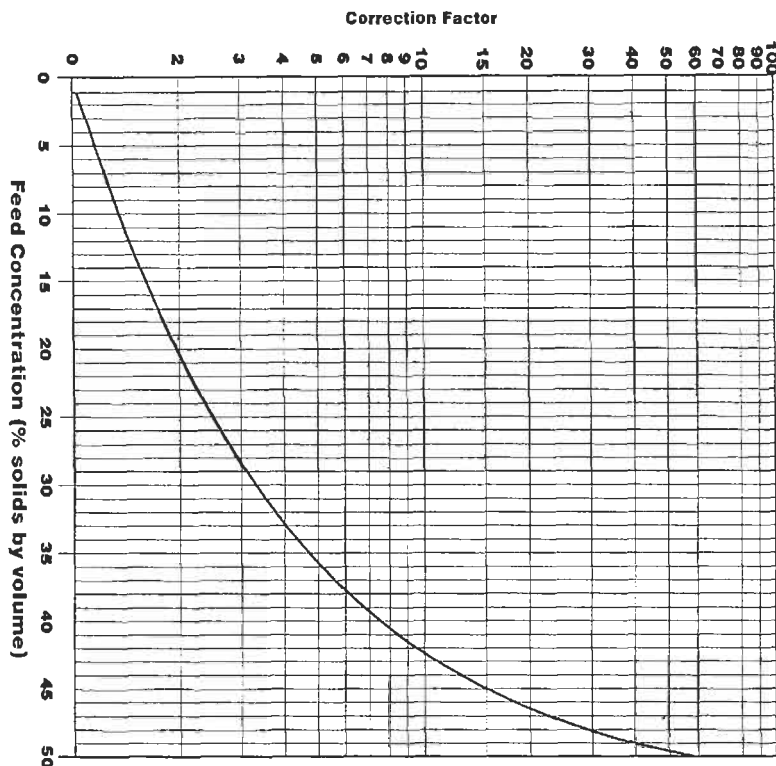
Figure 55. *Correction for feed concentration - after Afterburn.*

An analytical expression for the correction factor is as follows:

$$C_1 = [(53 - V)/53]^{-1.43}$$

where C_1 is the correction for the influence of the cyclone feed concentration and V is the percent solids by volume of cyclone feed. The second correction is for the influence of pressure drop across the hydrocyclone as measured by taking the difference between the feed pressure and the overflow pressure. Pressure drop is a measure of the energy being utilized in the cyclone to achieve the separation. It is recommended that pressure drops, whenever possible, be designed in the 40 to 70 kPa (5 to 10 psi) range to minimize energy requirements as well as reduce wear rates. This is especially true for coarse separations usually associated with primary or secondary grinding circuits.

The correction for pressure drop be calculated from the equation given below. This correlation indicates that a higher pressure drop would result in a finer separation and lower pressure drop in a coarser separation.

$$C_2 = 3.27 \times \Delta P^{-0.211}$$

where C_2 = Correction for influence of pressure drop.
            ΔP = Pressure drop in kPa.

The next correction is for the effect that specific gravity of the solids and liquid have on the separation. Since the cyclone does not actually achieve a size separation but rather a mass separation, the specific gravity of the particle is extremely important in determining the separation. It is especially meaningful in applications where the mineral has a higher specific gravity than the gangue material which allows better liberation of mineral particles at a coarser overall separation size. Stoke's law can be applied to determine particle diameters which would produce the same terminal settling velocity for a particle of known specific gravity in a liquid of known specific gravity as compared to a particle of 2.65 specific gravity in water. This relationship can be calculated using the following equation.

$$C_3 = [1.65/(\gamma_S - \gamma_L)]^{0.5}$$

where C_3 = Correction for influence of specific gravity
            γ_S = Specific gravity of solids
            γ_L = Specific gravity of the liquid

The cyclone diameter, along with the three corrections of percent solids, pressure drop, and specific gravity, are the main variables necessary for preliminary sizing and selection of cyclones. Other variables, such as the vortex finder and inlet size, also have an effect on separation. For example, a larger vortex finder size would tend to coarsen the separation, whereas a smaller size would tend to achieve a finer separation. Due to this fact, most cyclones have a replaceable vortex finder with different sizes available. Vortex finder diameters vary from a minimum of about 25% of the cyclone diameter to a maximum of about 45%. The inlet area also shows the same effect as the vortex finder, but not as pronounced. The apex size also has an effect on separation but the effect is minor unless the apex is too small and becomes a physical constraint, forcing material into the overflow. Cyclone retention time is also a minor factor influencing cyclone performance. Within limits, increased retention time would help achieve a finer separation; whereas reduced retention time would coarsen the separation. The retention time of the cyclone can be altered by either changing the length of the cylindrical section or by changing the cone angle. There are other variables which also have an effect of separation; however, these variables are relatively minor and may be neglected for the preliminary sizing and selection of cyclones.

Another important objective which must be considered is to provide adequate cyclone capacity for the application. The volume of feed slurry that a given cyclone can handle is related to the pressure drop across the cyclone. The relationship between flow rate and pressure drop for several different sizes of standard cyclones is shown in Figure 56. As shown, the flow rate increases as the pressure drop increases. In order to utilize this graph, the pressure drop used for calculating the separation is used to determine the flow rate for the cyclone diameter which was

also used for determining the separation. The flow rate is then divided into the total flow for a specific application to determine the number of units necessary. Since the flow rate given in Figure 56 is for water rather than slurry, it should be mentioned that slurry normally increases the capacity of a hydrocyclone over that shown for water; however, for preliminary estimates this factor can be neglected. This results in the number of cyclones calculated being slightly higher than those actually needed. Approximately 20% to 25% standby cyclones are recommended for operational as well as maintenance flexibility. The vortex finder size and inlet area of a cyclone also have an effect on the volumetric flow rate that a given cyclone can handle. Larger vortex finders or inlet areas would increase the capacity, whereas smaller vortex finders or inlet areas would decrease the capacity. The proper selection of apex size is critical to proper cyclone performance. For each application a circulating load is normally given which establishes the amount of solids which must pass through the cyclone underflow. Experience has shown that an underflow density of 50% to 53% solids by volume is typical for primary grinding circuits, whereas an underflow density of 40% to 45% solids by volume is normal for regrind circuits. Therefore, an underflow density can be assumed which establishes the total flow rate that must report through each cyclone apex. Figure 57 shows the approximate flow rate for a given diameter apex orifice. One of the most important considerations is to insure that cyclones are installed properly. A most important consideration is proper delivery of the slurry to the cyclone or cyclones (called multiclones). It has been found that a pipe size which produces a line velocity of 200 to 300 cm/sec (7 to 10 ft/sec) is high enough to prevent particles from settling, even in horizontal sections, but low enough to minimize wear. Normally for a single cyclone installation the inlet pipe size of the manufacturer's recommendation produces a velocity in this area. If the slurry is to be distributed to a number of cyclones operating in parallel, extreme care should be given to the design of the distribution system, and a radial type of manifold is recommended. This is a system where the cyclones are fed from a central circular chamber. When properly designed the central chamber becomes a mixing area and the line velocity should be lowered to approximately 60 to 90 cm/sec (2 to 3 ft/sec). This will help insure that each cyclone is fed with the same slurry concentration as well as the same particle size distribution and also will reduce wear rates. Using the radial manifold also makes it easier to install standby cyclones. Should an inline type manifold be utilized, the cyclones do not receive good distribution. It is typical that the high mass particles or coarser particles tend to pass the first cyclones and report to the final cyclone. This results in the last cyclone receiving a higher feed concentration of coarser particles, which accelerates the wear of the last cyclone as well as produces a coarser separation due to the higher feed density. Also, the last cyclone, once shut off, becomes difficult to restart because the solids will tend to pack into the feed pipe. For applications where the separation is not critical or one in which the feed concentration is extremely low, an inline manifold is acceptable and is much less expensive than the radial type.
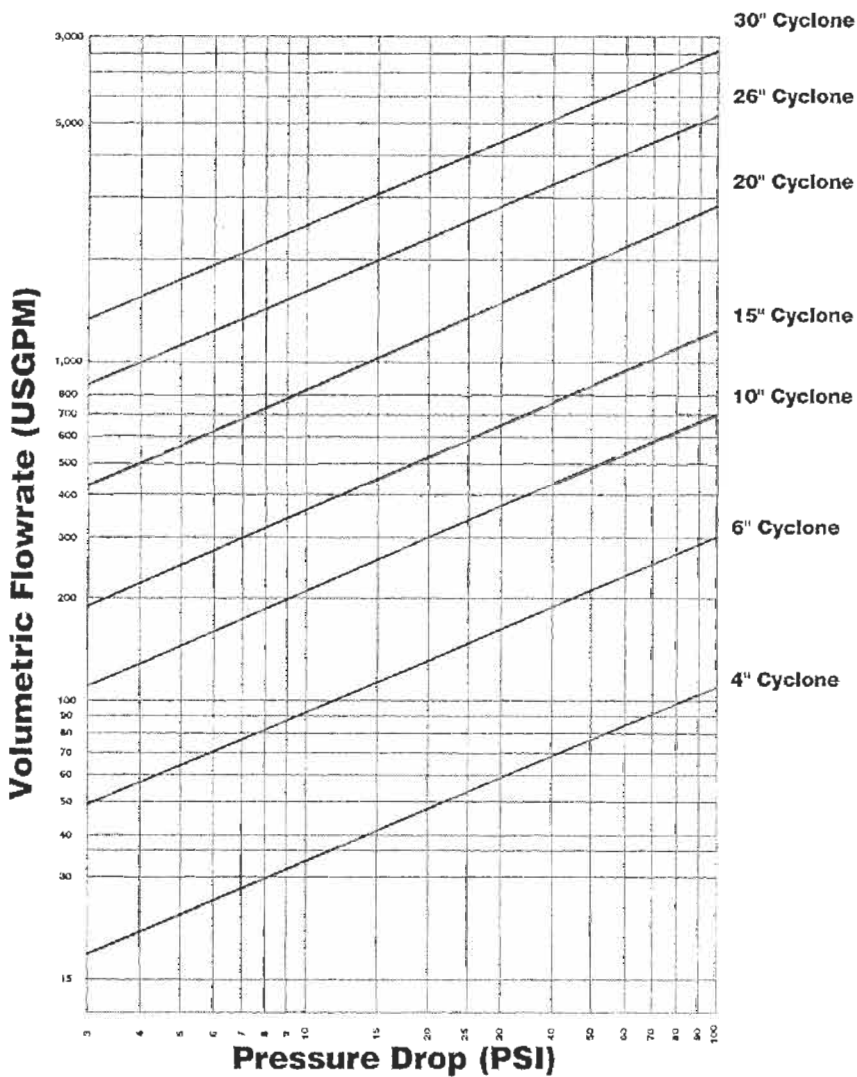
Figure 56. *Water capacity for standard cyclones, after Afterburn.*

Cyclone construction varies greatly from one manufacturer to another, but the majority of the designs includes metal housings with replaceable liners with replaceable liners with the most suitable liner material varying from one application to another. It is also common to utilize several different lining materials within the same cyclone to optimize wear characteristics. Natural gum rubber is the most common material utilized due to its relatively low cost, excellent wear, ease of handling, and it is not fragile. Applications where gum rubber is not suitable are those where the temperatures exceed 60 °C or there are large amounts of hydrocarbons present in the slurry such as oil or other detrimental chemicals.
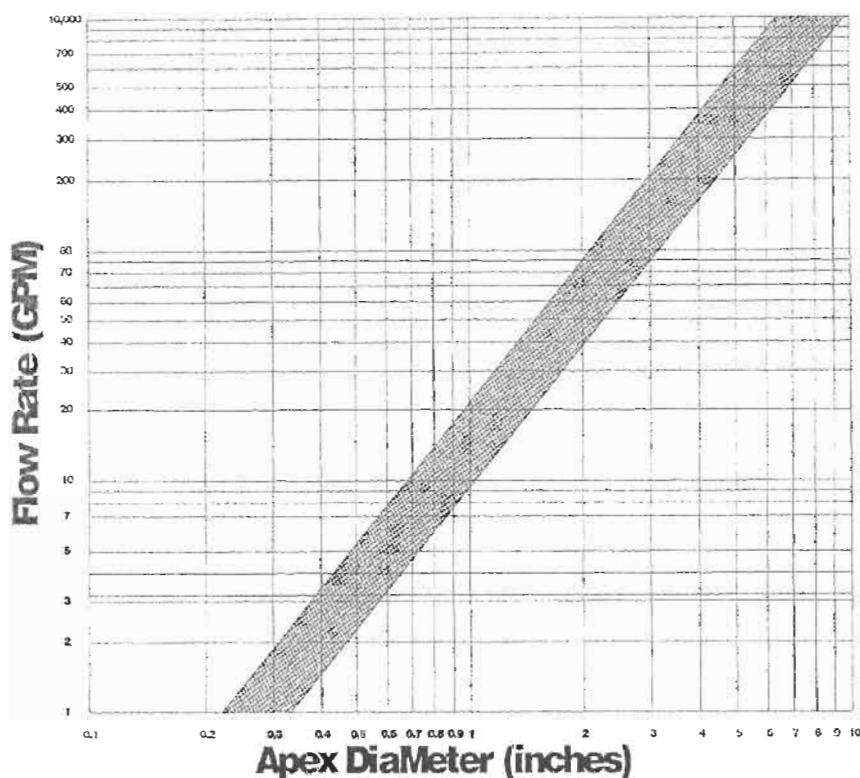
Figure 57. *Apex capacity diameter vs flowrate, after Afterburn.*

Ceramic materials have found acceptance as cyclone liners for the apex orifice as well as other areas which exhibit severe abrasion. These include areas such as the lower cone liner and vortex finder. Nihard has also proven to be an acceptable wear material, especially for vortex finders and other areas which require strength as well as abrasion resistance. Other elastomer materials such as neoprene and nitrite are also utilized when hydrocarbons are present or when the temperature exceeds 60 °C. Urethane has found acceptance, especially in areas where the solids are relatively fine.

A final note is with regard to flat bottom cyclones which have gained acceptance in recent years. The installation of a flat bottom in place of the conical section will coarsen the D_{50c} separation by more than twofold. Additionally, the sharpness of the recovery curve will decrease significantly. As such, flat bottom cyclones should be restricted to those applications in which coarse separations are required. The flat bottom cyclone does produce a very clean underflow but at the expense of a large amount of misplace coarse solids in the overflow. An illustration of a flat bottom cyclone is shown in Figure 58. The reader should refer to the reference section of this chapter for citations that provide more in-depth coverage of this equipment, as well as design case studies and example.

90° CONE ANGLE
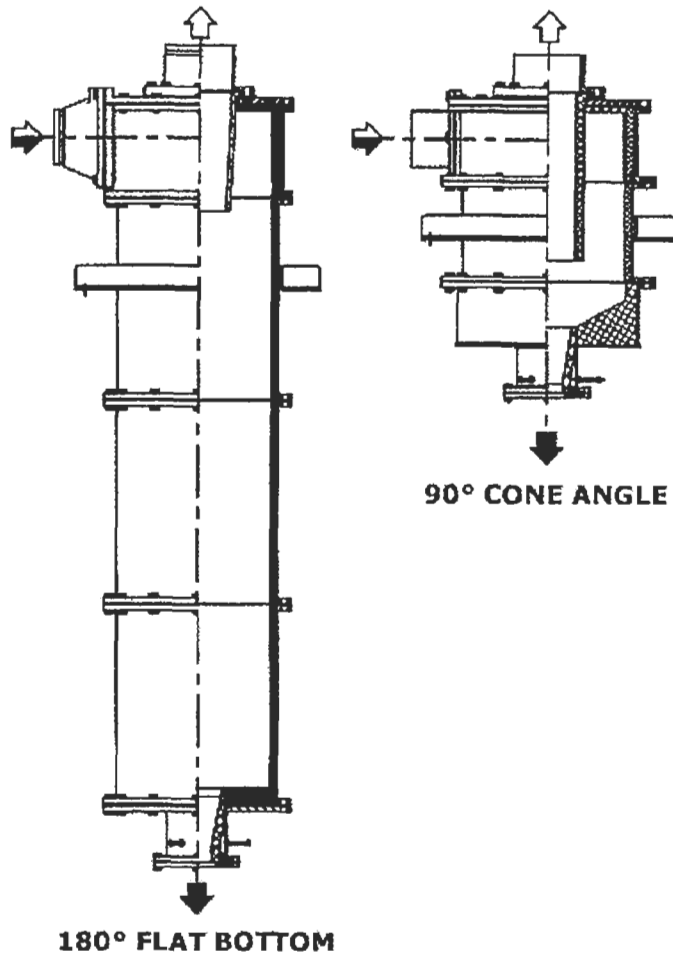
180° FLAT BOTTOM

Figure 58. *Examples of flat bottom cyclones.*

Sedimentation Centrifuges

Another method to the separation of solids from suspensions in a centrifugal field is through the use of sedimentation centrifuges. As in the hydroclone, particles of the heavier phase fall through the lighter phase away from the center of rotation. In centrifuges, liquid (or gas) and solids are acted on by two forces: gravity acting downward and centrifugal force acting horizontally. In industrial units, however, the centrifugal force component is normally so large that the gravitational component may be neglected. The magnitude of the centrifugal force component is defined by the ratio $R_c/G = \omega^2 r/g$, which is referred to as the "relative centrifugal force" (RCF) or *centrifugal number,* N_c, where $R_c = m\omega^2/2$ or $R_c =$

$mv^2/2$ is the centrifugal force, and $F_g = mg$ is the gravitational force. The RCF typically varies from 200 times gravity for large-basket centrifuges to 360,000 for high-speed tubular-gas centrifuges and ultracentrifuges. For liquid-solid separations, centrifugal force may be applied in sedimentation-type centrifuges, centrifugal filters or in a combination of both. Sedimentation-type centrifuges also are used for size or density classification of solids, the separation of immiscible liquids of different densities and for concentrating gases of different molecular weights. The principle actions of centrifugation are as follows. A stationary cylindrical bowl contains a suspension of solid particles in which the particle density is greater than that of the liquid. Because the bowl is stationary, the free liquid surface is horizontal and the particles settle due to the influence of gravity. Once the bowl is rotating about its vertical axis, liquid and solid particles are acted on by gravity and centrifugal forces, resulting in the liquid assuming a position with an almost vertical inner surface (free interface). If the suspension consists of several components, each with different densities, they will stratify with the lightest component nearest the axis of rotation and the heaviest adjacent to the solid bowl wall. The bowl wall is perforated and lined with a permeable membrane, such as filter cloth or wire screen, which will support and retain the solid particles but allow the liquid to pass through due to the action of centrifugal force.

The main components of a centrifuge are a rotor or bowl in which the centrifugal force is applied to a heterogeneous system to be separated; a means for feeding this system into the rotor; a drive shaft; axial and thrust bearings; a drive mechanism to rotate the shaft and bowl; a casing or "covers" to contain the separated components; and a frame for support and alignment. There are three main types of centrifuges, which may be classified according to the centrifugal number, and the range of throughputs or the solids concentration in suspension that can be handled. The first of these is the tubular-bowl centrifuge. This type has a centrifugal number in the range of 13,000 but is designed for low capacities (50 to 500 gph) and can handle only small concentrations of solids.

The second is the *solid-bowl centrifuge,* with maximum bowl diameters ranging from 4 to 54 inches. The larger diameter machines can handle up to 50 ton/hr of solids with a centrifugal number up to 3,000. Similar centrifuges are manufactured with a perforated wall on the bowl. These machines operate exactly like filters, with the filtrate draining through the cake and bowl wall into a surrounding collector.

The third type is the *disk-bowl centrifuge,* which is larger than the tubular-bowl centrifuge and rotates at slower speeds with a centrifugal number up to 14,000. These machines can handle as much as 30,000 gph of feed containing moderate quantities of solid particles. The migration of particles in sedimentation centrifuges is radially toward or away from the axis of rotation, depending on whether the density of the dispersed particles is greater, or less, than that of the continuous phase.

There must be a measurable difference between the density of the continuous and dispersed phases to provide effective separation. In commercial machines the

discharge of the liquid, or separated liquid phases is performed almost always in a continuous fashion. The heavy solid phase deposited against the bowl wall is discharged and recovered intermittently, manually, or by action of an unloader knife or skimmer; continuously by action of a differential screw conveyor; or intermittently or continuously with a portion of the continuous phase through openings in the wall of the bowl. Manual solids removal units can operate continuously up to one hour and generally only require a few seconds for a fully automated intermittent operation. In systems in which solids have a lesser density than the continuous phase, particulates can be removed continuously from the surface of the liquid via a skimming tube or exit as an overflow from the bowl with a portion of the continuous phase.

Tubular-bowl centrifuges are used extensively for the purification of oils by separating suspended solids and free moisture from them; for removal of oversize particles from dye pastes, pigmented lacquers and enamels; for "polishing" citrus and other aromatic oils; and other small-scale separating applications. The tubular-bowl clarifiers and separators are comprised of small-diameter cylinders (about 100 mm) which allows operation at very high velocities. Commercial machines typically work at 15,000-19,000 rpm, which corresponds to N_c = 13,000 to 18,000. For special applications (e.g., treatment of vaccines, etc.), the diameter of the tubular-bowl centrifuge is only several centimeters, with values of N_c as high as 50,000. The tubular centrifuge rotor is suspended from its drive assembly on a spindle that has a built-in degree of flexibility. It essentially hangs freely with a sleeve bushing in a dampening assembly at the bottom. In some designs a similar dampening assembly is included at the upper end of the rotor. This permits the rotor to determine its own mass axis after it exceeds its critical speed. The feed liquid is introduced to the rotor at the bottom through a stationary feed nozzle. The inlet feed is under sufficient pressure to create a standing jet, which ensures a clean entrance into the rotor. Often, an acceleration device is provided at the bottom of the rotor to bring the feed stream to the rotational speed of the bowl. The feed moves upward through the bowl as an annulus and discharges at the top. To effect this, the radius of the discharge at the top must be larger than the opening at the bottom through which the feed enters. Solids move upward with the velocity of the annulus and simultaneously receive a radial velocity that is a function of their equivalent spherical diameter, their relative density, and the applied centrifugal force. If the trajectory of a particle intersects the cylindrical bowl wall, it is removed from the liquid; if it does not, the particle flows out with the effluent overflow.

Multichamber (multipass) centrifuges combine the process principles of a tubular clarifier with mechanical drive and the bowl contour of a disk centrifuge. The suspension flows through a series of nested cylinders of progressively increasing diameter. The direction of the flow from the smallest to the largest cylinders is in parallel to the axis of rotation, as in the tubular bowl. The rotor usually contains six annuluses, so that the effective length of suspension travel is approximately six times the interior height of the bowl. The multipass bowl can be considered as a

multistage classifier because the centrifugal force acting in the machine is greater in each subsequent annulus. Consequently, larger, heavier particles are deposited in the first annulus (the zone of least centrifugal force), while smaller, lighter particles are deposited in the last annulus (the zone of greatest centrifugal force). The radial distance particles must migrate to reach the cylinder wall is thus minimized. Multipass rotors typically have a total holding volume of up to 65 liters for the largest size, of which about 50% is available for the retention of collected solids before the clarification process is impaired. As with the tubular type, the collected solids must be removed manually. These machines are applied to the clarification of fruit and vegetable juices, wine, and beer.

The continuous solid-bowl centrifuge consists of a solid-wall rotor, which may be tubular or conical in shape, or a combination of the two. The rotor may rotate about a horizontal or a vertical axis because the centrifugal force is many times that of gravitational force (for many units N_c is more than 3,000). An example of this kind of equipment is a continuous horizontal centrifuge, as shown in Figure 59. It consists of a cylindrical rotor with a truncated cone-shaped end and an internal screw conveyor rotating together. The screw conveyor often rotates at a rate of 1 or 2 rpm below the rotor's rate of rotation. The suspension enters the bowl axially through the feed tube to a feed accelerated zone, then passes through a feed port in the conveyor hub into the pond. The suspension is subjected to centrifugal force and thrown against the bowl wall where the solids are separated. The clarified suspension moves toward the broad part of the bowl to be discharged through a port.

The solid particles being scraped by the screw conveyor are carried in the opposite direction (to the small end of the bowl) across discharge ports through which they are ejected continuously by centrifugal force. As in any sedimentation centrifuge, the separation takes place in two stages: settling, and thickening or pressing out of the sediment. Because the radius of the solid discharge port is usually less than the radius of the liquid overflow at the broader end of the bowl, part of the settled solids is submerged in the pond. The remainder, closer to the center, is inside the free liquid interface, where they can drain before being discharged.
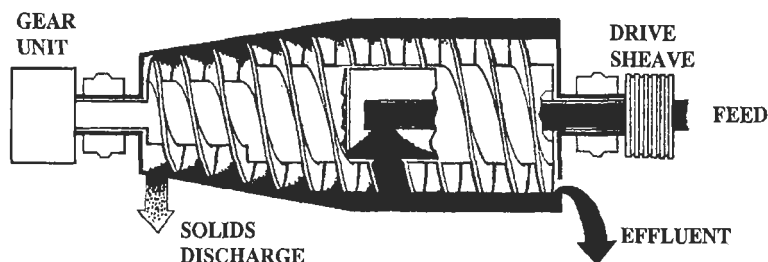
Figure 59. *Continuous solid-bowl centrifuge.*

The total length of the "settling" and "pressing out" zones depends on the dimensions of the rotor. Their relative length can be varied by changing the pond level through suitable adjustment of the liquid discharge radius. When the pond depth is lowered, the length of the pressing out zone increases with some sacrifice in the clarification effectiveness.

The critical point in the transport of solids to the bowl wall is their transition across the free liquid interface, where the buoyancy effect of the continuous phase is lost. At this point, soft amorphous solids tend to flow back into the pond instead of discharging. This tendency can be overcome by raising the pond level so that its radius is equal to, or less than, that of the solids discharge port. In reality, there are no dry settled solids. The solids form a dam, which prevents the liquid from overflowing. The transfer of solids becomes possible because of the difference between the rotational speed of the screw conveyor and that of the bowl shell. The flights of the screw move through the settled solids and cause the solids to advance. To achieve this motion, it is necessary to have a high circumferential coefficient of friction on the solid particles with respect to the bowl shell and a low coefficient axially with respect to the bowl shell and across the conveyor flights. These criteria may be achieved by constructing the shell with conical grooves or ribs and by polishing the conveyor flights. The conveyor or differential speed is normally in the range of 0.8% to 5% of the bowl's rotational speed.

The required differential is achieved by a two-stage planetary gear box. The gear box housing carrying two ring gears is fixed to, and rotates with the bowl shell. The first stage pinion is located on a shaft that projects outward from the housing. This arrangement provides a signal that is proportional to the torque imposed by the conveyor. If the shaft is held rotational (for example, by a torque overload release device or a shear pin), the relative conveyor speed is equivalent to the bowl rotative speed divided by the gear box ratio. Variable differential speeds can be obtained by driving the pinion shaft with an auxiliary power supply or by allowing it to slip forward against a controlled breaking action. Both arrangements are employed when processing soft solids or when maximum retention times are needed on the pressing out zone. The solids handling capacity of this type centrifuge is established by the diameter of the bowl, the conveyor's pitch and its differential speed.

Feed ports are located as far from the effluent discharge as possible to maximize the effective clarifying length. Note that the feed must be introduced into the pond to minimize disturbance and resuspension of the previously sedimented solids. As a general rule, the preferred feed location is near the intercept of the conical and cylindrical portions of the bowl shell. The angle of the sedimentation section with respect to the axis of rotation is typically in the range of 3 to 15°. A shallow angle provides a longer sedimentation area with a sacrifice in the effective length for clarification. In some designs, a portion of the conveyor flights in the sedimentation area is shrouded (as with a cone) to prevent intermixing of the sedimented solids with the free supernatant liquid in the pond through which they normally would pass. In other designs, the clarified liquid is discharged from the front end via a

centrifugal pump or an adjustable skimmer that sometimes is used to control the pond level in the bowl. Some displacement of the adhering virgin liquor can be accomplished by washing the solids retained on the settled layer, particularly if the solids have a high degree of permeability. Washing efficiency ranges up to 90% displacement of virgin liquor on coarse solids. The longer section of a dry shallow layer provides more time for drainage of the washed solids. This system is especially effective for washing and dewatering such spherical particles as polystyrene. In either washing system, the wash liquid that is not carried out with the solids fraction returns to the pond and eventually discharges along with the effluent virgin liquor.

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Chapter 7
MIXING EQUIPMENT

INTRODUCTION

Mixing is a unit operation that is practiced widely to meet a variety of process requirements. The specific mixing system design, operating arrangement and power requirements depend largely on the desired form of the intermediate or final products. Mixing is applied to achieve specified results in the following situations: creating a suspension of solid particles; the blending miscible liquids; dispersing gases through liquids; blending or dispersing immiscible liquids in each other; and promoting heat transfer between a fluid (liquid) and the coil or jacket of a heat exchanging device. The operating characteristics and design configuration of a mixing system are established on the basis of the required energy expenditure to create or approximate a homogeneous fluid system. For example, in producing an emulsion one must supply sufficient energy to "break up" the dispersed phase. In doing so, high shear stresses, which depend on velocity gradients, are developed in the mixing medium. In the zones in which the velocity gradient approaches a maximum, an intensive breaking up of the dispersed phase occurs. Mixing reduces concentration and temperature gradients in the processed system, thus exerting a favorable effect on the overall rates of mass and heat transfers. This applies in particular to dissolving applications, electrolysis, crystallization, absorption, extraction, heating or cooling, and heterogeneous chemical reactions, which proceed for the greater part in a liquid medium. Increased turbulence of the fluid system caused by mixing leads to a, decrease in the fluid's boundary layer thickness. This is derived from a continuous renewal of the surface contact area, resulting in a pronounced rate of increase in heat and mass transfer mechanisms. Regardless which medium is mixed with the liquid, i.e., gas, liquid or solid particles, two basic methods are employed. These arc mechanical mixers, which utilize different types of impellers, and pneumatic mixers, which utilize air or an inert gas to effect mixing. In addition to these designs, mixing also is achieved in normal fluid handling operations, such as in pumps and jet flows.

Two major characteristics of all mixing devices that provide a basis for comparative evaluations are: (1) the efficiency of a mixing device, and (2) the intensity of mixing. The *efficiency* of a mixing device characterizes the quality of the process to be treated and may be expressed differently depending on the mixing purpose. For example, in producing suspensions mixing efficiency is characterized by the uniform distribution of the solid phase in the volume of equipment. For the intensification of thermal and diffusion processes, it is characterized by the ratio of

mixed and unmixed heat and mass transfer coefficients, respectively. Mixing efficiency depends not only on the equipment design, but also on the amount of energy introduced in the liquid being agitated.

The intensity of mixing is determined by the time required to achieve a desired technological result or by the mixer rpm at fixed process conditions (for mechanical mixers). From an economical standpoint, it is beneficial to achieve the required mixing effect in the shortest possible time. In evaluating the energy required for a mixing operation, one must account for the total energy consumption during the time needed to achieve a specified mixing result.

Mechanical mixers, which for the most part comprise rotating devices, are employed for liquids almost exclusively.

MECHANICAL MIXING EQUIPMENT

Mechanical mixing devices comprise three basic parts: an impeller, a shaft and a speed-reducing gearbox. The impeller constitutes the working element of the apparatus, mounted on a vertical, horizontal, or inclined shaft. The drive may be connected directly to a motor or through a gear box. The multitude of impeller configurations can be grouped into five distinct categories, of which only the first four are of major industrial importance. Discussions to follow concern only the three most widely used types, namely propeller, turbine and paddle mixers. The two basic propeller mixer configurations are fixed to a rotating vertical, horizontal or inclined shaft. The first is similar to an aircraft propeller, while the second resembles a marine propeller. Depending on the height of liquid layer, one shaft may carry one to three propellers. Due to their more streamlined shape, a propeller mixer's power requirements are less than the other types of mixers at the same Reynolds number. Their transition in the self-modeling region is observed at relatively low values of Reynolds number, $Re \approx 10^4$. They are capable of high-speed operation without the use of a gearbox and, hence, provide a more cost-effective operation because there are no mechanical losses in transmission. Propeller mixers produce an axial flow, which has a great pumping effect and provides short mixing times. Disadvantages compared to paddle and turbine mixers are higher cost, the sensitivity of operation to the vessel geometry and its location within the tank. As a general rule, propeller mixers are installed with convex bottom vessels. They should not be used in square tanks or in vessels with flat or concave bottoms.

A rotating propeller traces out a helix in the fluid, from which a full revolution moves the liquid longitudinally to a fixed distance, depending on its pitch, i.e., the ratio of this distance to the propeller diameter. Pitch may be computed from the following formula:

$$s = 2\pi r \tan(\psi)$$

where r is the propeller blade radius and, therefore, also the radius of the cylinder

created in the liquid as derived from the movement of the impeller, and ψ is the angle of tilt of the blade. Pumping and mixing efficiencies increase with pitch, as achieved by the axial flow of the liquid from the impeller. This flow results from the delivery head of the propeller and the helical turbulent flow of the entire contents of the vessel, which is caused by the radial velocity gradients in the liquid strata at different distances from the impeller. At high rotational speeds the entire fluid mass swirls despite the axial flow, and a central vortex begins to form around the shaft.

Draft tubes are employed to improve the mixing of large quantities of liquids by directing the motion of the liquid. Figure 1 shows such an arrangement, favorable for large ratios of liquid depth to mixer diameter. In such applications a high pumping capacity of the mixer is utilized, especially where mixtures of low viscosity are concerned. The draft tube directs the flow to the regions of the vessel that otherwise would not be agitated by the liquid stream. In the absence of draft tubes and at high rotational velocities of the propeller, baffles generally are located at various points in the vessel. Baffles minimize vortex formation and divide it into a number of local eddies, increasing the total turbulence of the tank. Depending on the application, multiple impellers may be mounted on a single revolving shaft and more than one shaft may be employed in a given tank. In some applications it is desirable to have two adjacent impellers rotating in opposite directions, forming a beater. Sometimes the impellers actually touch the walls of the tank, giving a positive scraping action, which is desirable when thick layers of material tend to stick to the wall.

Propeller mixers are used for mixing liquids with viscosities up to 2,000 cp. They are suitable for the formation of low-viscosity emulsions, for dissolving applications and for liquid-phase chemical reactions. For suspensions, the upper limit of particle size is 0.1 to 0.5 mm, with a maximum dry residue of 10%.
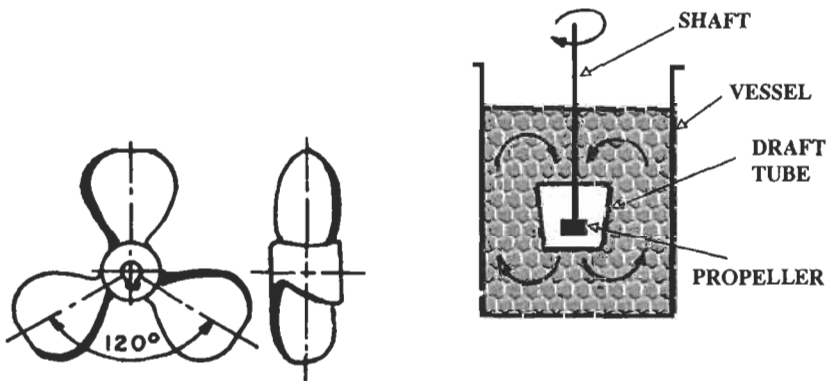
Figure 1. *Typical propeller mixer configuration.*
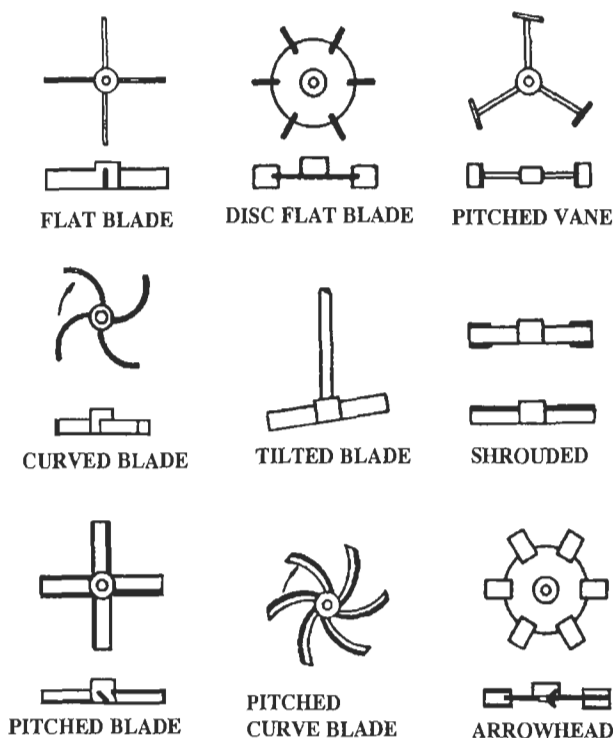
Figure 2. *Turbine impeller configurations.*

Propeller mixers are unsuitable for suspending rapid settling substances and for the absorption of gases. Propellers are designed on the basis of data obtained from properly executed modeling experiments.

The turbine mixer represents a very important class. A turbine mixer as an impeller with essentially a constant blade angle with respect to a vertical plane, over its entire length or over finite sections, having blades either vertical or set at an angle less than 90° with the vertical. Blades may be curved or flat, as shown by the various configurations in Figure 2. Turbine mixer operation is analogous to that of a centrifugal pump working in a vessel against negligible back pressure. The mixing action is accomplished by the turbine blades, which entrain and discharge the liquid.

The predominantly radial flow from the impeller impinges onto the vessel walls, where it splits into two streams. These streams cause mixing by their energy. When turbine mixers are operated at sufficiently high rotational speeds, both radial and tangential flows become pronounced, along with vortex formation. This flow situation warrants the installation of baffles to ensure a more uniform flow distribution throughout the mixing vessel.
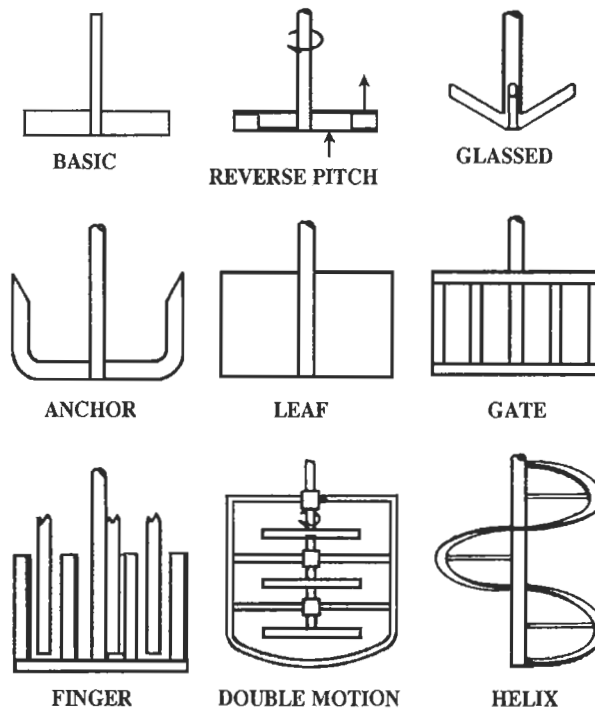
Figure 3. *Paddle mixer configurations.*

Paddle Mixers

Paddle mixers are devices consisting of two or more blades mounted on a vertical or inclined shaft. The basic paddle impeller configurations are shown in Figure 3. The main advantages of paddle mixers are their simplicity and low cost. A disadvantage is their small pumping capacity (a slow axial flow), which does not provide a thorough mixing of the tank volume. Perfect mixing is attained only in a relatively thin stratum of liquid in the immediate vicinity of the blades. The turbulence spreads outward very slowly and imperfectly into the entire contents of the tank; hence, circulation of the liquid is slow. Therefore, paddle mixers are used for liquids with viscosities only up to about 1,000 cp. Because of a concentration gradient that often is created in the liquid when these type mixers are used, they are unsuitable for continuous operation. This can be remedied by tilting the paddle blades 30 - 45° to the axis of the shaft, resulting in an increase in axial flow and, consequently, a decrease in concentration gradients. Such a mixer can maintain particles suspended, provided settling velocities are not high. Mixers with tilted blades are used for processing slow chemical reactions, which are not limited by

diffusion. To increase the turbulence of the medium in tanks with a large height to diameter ratio, a configuration is employed that consists of several paddles mounted one above the other on a single shaft. The separation between individual paddles lies in the range of 0.3 to 0.8 d (where d is the diameter of the paddle) and is selected according to the viscosity of the mixture. For mixing liquids with viscosities up to 1,000 cp, as well as for heated tanks in cases in which sedimentation can occur, anchor or gate paddle mixers are employed (refer to Figure 3). Paddle diameters are almost as large as the inside diameter of the tank in such applications, so that the outer and bottom edges of the paddle scrape (or clean) the walls and bottom.

Leaf-shaped (broad blade) paddle mixers provide a predominant tangential flow of liquid, but there is also turbulence at the upper and lower edges of the blade. Leaf-type blades are employed for mixing low-viscosity liquids, intensifying heat transfer processes, promoting chemical reactions in a reactor vessel and for dissolving materials. For dissolving applications, leaf blades usually are perforated. During the mixer's rotation, jets are formed at the exits from the holes which promotes the dissolution of materials. The rotational velocity of paddle mixers is in the range of 15 to 45 rpm. Under these conditions. the pumping action is small and there generally is no danger of vortex formation. As such, paddle mixers are most often used in vessels without baffles. However, for broad-blade paddles, which operate at speeds up to 120 rpm, baffles are incorporated into the design to minimize vortex formation.

Emulsifiers, Blenders and Planetary Mixers

There are a number of other mixing configurations that have widespread applications to both liquid-solid and solid-solid blending operations. Emulsifiers are used as an alternative to slow-speed impeller mixing or high-pressure homogenization for a wide range of processing requirements. Typical applications include the preparation of adhesives (e.g., asphalts, carbon dispersions, clay dispersions, dyestuffs, paints and inks, lacquers), cosmetics (e.g., creams, emulsions, hand lotions, perfumes, shampoos, deodorants), foods (e.g., chocolate coatings, mustard, soft drinks, sugar emulsions), pharmaceuticals (e.g., antibiotics, ointments, reducing animal tissues), plastics (e.g., cold cutting resins, polyester dispersions, resin solutions) and various miscellaneous mixtures such as floor polishes, gum dispersions, lubricants, petroleum emulsions, etc. These types of mixers normally are used in dished or conical bottom vessels. The mixing process can be thought of as performed in three stages. In the first stage, the high-speed rotor operating at close clearance to the stator draws material in from the bottom of the mixing vessel and subjects it to intense mixing. In stage 2, the rotor accelerates the product toward the blade periphery. There it is expelled through openings in the stator into the body of the mix while undergoing an intensive mechanical and hydraulic shearing action. At the same time, new material is drawn

into the center of the rotor. In the third stage, the expelled mixture is deflected by the tank wall, completing the circulation. To increase circulation or to create a vortex for the incorporation of light solids, a downthrust propeller may be mounted on the rotor shaft.

The operating sequence for emulsifiers varies with the intended application. Standard batch units are available through 100 hp. For most applications the rotor head should be mounted two to three head diameters above the vessel floor. Obviously, with these designs a slender-shaped vessel generally is preferred. Maximum mixing efficiency generally is achieved on mixtures under 10,000 cp. The principal criterion for achieving the best efficiency is to maintain a maximum circulation of all materials through the rotor/stator at all times during the process cycle. As viscosity increases, flow through the head decreases, thus lessening the work on a given volume of material while it is being circulated within the vessel. Inline emulsifiers are less susceptible to this problem as they may be fed by means of positive displacement pumps. Note that because of the wide range of applications to which a single design is subjected, the density and rheology of the mixture are variables. The pumping capabilities of a unit on materials that have the same apparent viscosity may not be the same in actual practice. For example, many polymers have a low apparent viscosity and appear ideally suited to a particular unit design and application criteria. However, on testing, it may be observed that the flow is poor and that the end result is only marginal. Often, modified tank configurations, auxiliary agitation, different rotor speeds or head diameters will improve efficiency. Pilot testing almost always is recommended for accurate scaleup.

Ribbon blenders resemble helical paddle mixers both in design and operation, the principal difference being that the shaft orientation is strictly horizontal. There are basically three standard agitator designs, namely, continuous ribbon, interrupted ribbon, and paddle type. They may be arranged for either center or end discharge from the mixing vessel. The principal designs are shown in Figure 4. The continuous ribbon, arranged for center discharge, produces homogeneous blends relatively quickly. The outer ribbons move materials toward the center, whereas inner ribbons move materials toward end plates. Both provide radial movement of materials. In the case of continuous ribbons, the outer ribbons move materials towards the discharge end of the vessel. The inner ribbons move materials in the opposite direction. Both provide radial transfer of materials. The interrupted ribbon provides the same basic action as a continuous ribbon agitator. It requires less power, and generally is applied to materials with high bulk densities. The paddle-type is often used in such applications as in the preparation of instant beverage premixes.

Ribbon blenders are essentially self-contained mixers. They are employed in a variety of solid-liquid, solid-solid, and liquid-liquid blending applications in the chemical process industries. Examples include plastics, pigments, pharmaceuticals, specialty chemicals, confectionary.
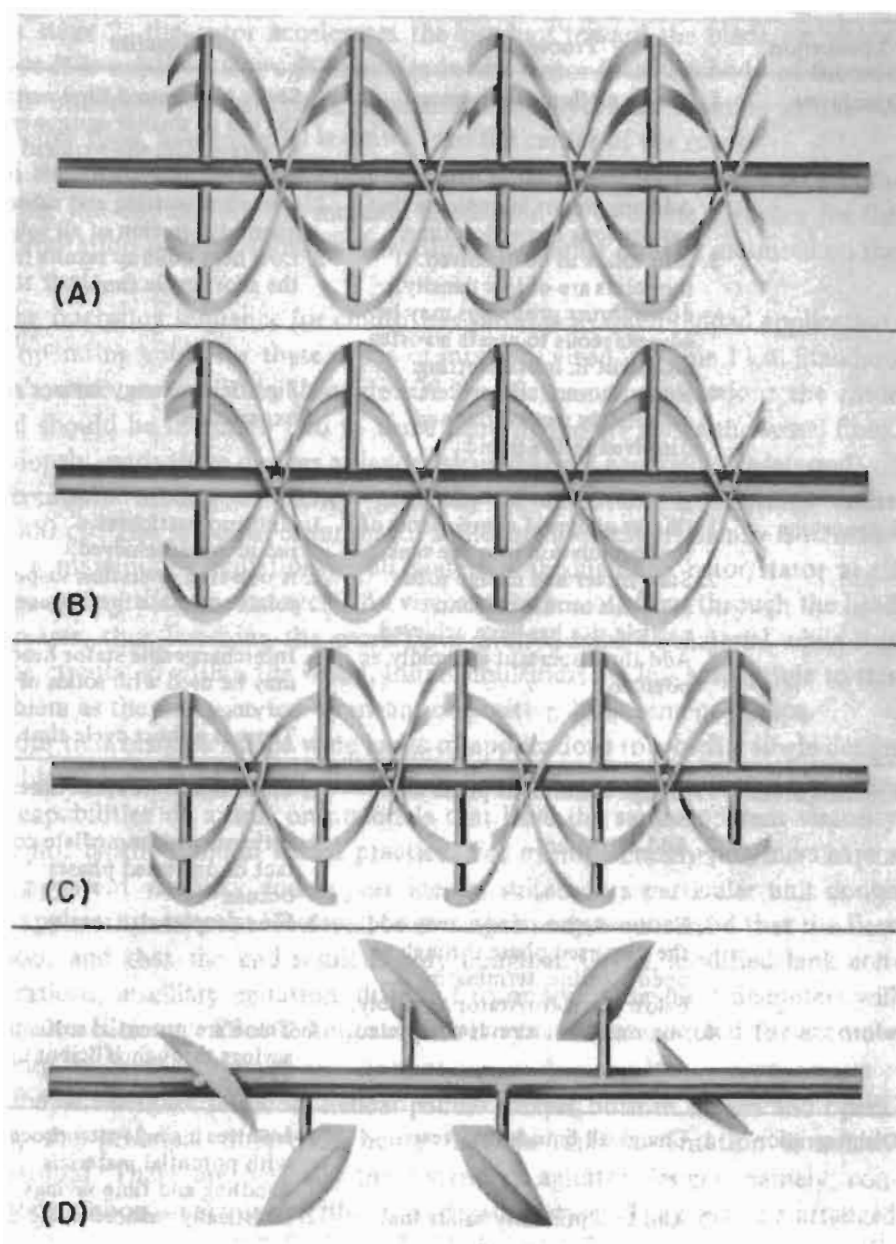
Figure 4. *Ribbon blender agitator configurations:(A) continuous ribbon, arranged for center discharge; (B) continuous ribbon arranged for end discharge; (C) interrupted ribbon; (D) paddle-type ribbon.*

The reader may consult the reference section of this chapter for citations dealing with these types of agitator configurations and specific applications.

Planetary mixers are used for a variety of liquid and solid mixing applications, from simple mixtures to sophisticated reactions, involving high temperature, vacuum or internal pressure. This type of mixer is employed in batch operations. During the mix cycle, two rectangular-shaped stirrer blades revolve around the tank on a central axis. Each blade revolves on its own axis simultaneously, at approximately the speed of the central rotation. With each revolution on its own axis, each stirrer blade advances forward along the tank wall. This movement provides homogeneity of the material being mixed and does not depend on the flow characteristics of the mix. Instead, the stirrers cover every point within the mix tank. Double planetary mixers have no packing glands or bearings in the product zone. Hence, cleaning between batches is minimized. Figure 5 shows a planetary mixer unit. The machine is equipped with a hydraulic lift that permits the stirrer blades to be lowered and raised in and out of the mixing tank. The unit shown is capable of handling low-viscosity fluids to very high- viscosity pastes and doughlike materials. Typical capacities of these units range from 10 to more than 300 gallons.

Figure 5. *Planetary mixer with a vacuum hood.*

Rubber Compounding Equipment

Synthetic rubber or elastomers, in its raw state is too plastic for most commercial applications. Through a curing process termed vulcanizing, raw rubber can be made to lose plasticity and gain elasticity. By compounding the raw or "neat" rubber with various types and amounts of additives before the vulcanizing, tensile strength, abrasion resistance, resiliency, heat aging, and other desirable properties can be imparted to the rubber. The proportions and types of additives (including vulcanizing agents) compounded into the raw rubber, and the vulcanizing temperature, pressure, and time are varied in accordance with the properties desired in the final product. After the rubber is compounded, it is formed into the desired shape and then cured at the required temperature. In the forming steps, large amounts of organic solvents are often used in the form of rubber adhesives. Types of additives that are compounded into the rubber may be classified as vulcanizing agents, vulcanizing accelerators, accelerator activators, retarders, antioxidants, pigments, plasticizers and softeners, and fillers. Vulcanizing agents include peroxides and sulfur. Vulcanizing accelerators include aldehyde-amines, guanidines, and thiuram sulfides which are used to decrease the time and temperature required for vulcanization. Accelerator activators include zinc oxide, stearic acid, litharge, magnesium oxide, and amines which supplement the accelerators and, in addition, modify the finished product characteristics; for example, they increase the modules of elasticity. Examples of retarders include salicylic acid, benzoic acid, and phathalic anhydride to retard the rate of vulcanization. Antioxidants include many organic compounds, mostly alkylated amines, which are used to retard deterioration of the rubber caused by oxidation and improve aging and flexing ability. Pigments include such ingredients as carbon black, zinc oxide, magnesium carbonate, and certain clays which are used to increase tensile strength, abrasion resistance, and tear resistance. Iron oxide, titanium oxide, and organic dyestuffs are used to color the rubber. Plasticizers and softeners include resins, vegetable and mineral oils, and waxes which are used to improve resiliency, flexibility, and mixing and processing characteristics. Fillers include whiting, slate flour, barytes, and some of the pigments previously mentioned are used to improve processing properties and lower the cost of the finished product. In the compounding of blends, the accelerators are added first to the mass of raw rubber being milled or mixed. Then a portion of the plasticizers (if present in the blend recipe) are added, followed by the reinforcing pigments, the remainder of the plasticizers, the antioxidants, and any inert fillers or coloring agents. The vulcanizing agent is usually introduced as the last ingredient.

In order to be effective in imparting various chosen characteristics, all additives employed in a blend must be homogeneously dispersed throughout the blend. The two most commonly employed pieces of equipment for blending rubber and additives are rubber mills and a special type of mechanical mixer known as the Banbury mixer. A typical rubber mill consists of two rolls which rotate toward each

other at different speeds , creating a shearing and mixing action. Neat rubber is placed in the mill, and the additives are introduced, generally one or two components at a time. Additives may be finely divided solids or liquids. Another device commonly used for compounding rubber stock is the Banbury mixer.

Figure 6 shows cross-sections of two typical Banbury mixers. Each consists of a completely enclosed mixing chamber in which two spiral-shaped rotors, revolving in opposite directions and at different speeds, operate to keep the stock in constant circulation. A ridge between the two cylindrical chamber sections forces intermixing, and the close tolerances of the rotors with the chamber walls results in a shearing action. A floating weight in the feed neck confines the batch within the sphere of mixing. This combination of forces produces an ideally homogeneous batch.
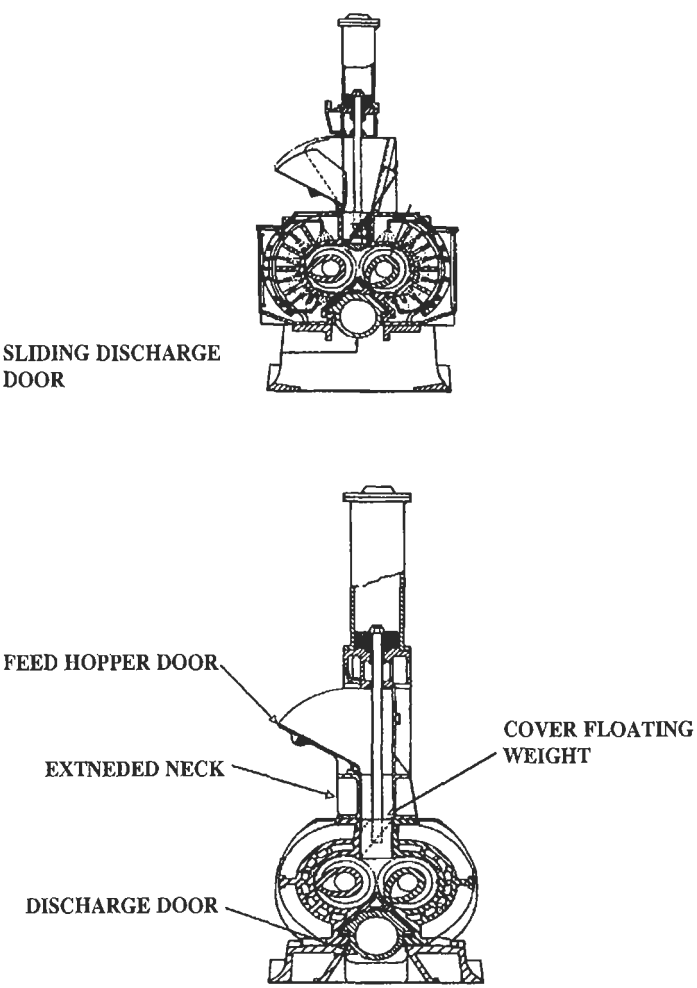
Figure 6. *Banbury mixer units.*

Flow Patterns in Agitated Tanks

The forces applied by an impeller to the material contained in a vessel produce characteristic flow patterns that depend on the impeller geometry, properties of the fluid, and the relative sizes and proportions of the tank, baffles and impeller. There are three principal types of flow patterns: tangential, radial and axial. Tangential flow is observed when the liquid flows parallel to the path described by the mixer as illustrated in Figure 7.

When the flow pattern in a mixed tank is primarily tangential, the fluid discharge from the impeller to the surroundings and its entrainment into the impeller are small. Also, fluid transfer in the vertical direction is at a minimum. The mixing effect is lowest when the rotational velocity of the liquid approaches that of the mixer.

With radial flow, the liquid discharges from the impeller at right angles to the mixer axis and moves along the radius. As soon as the centrifugal force, which depends on the impeller's diameter and speed, overcomes the resistance of the medium, it initiates radial flow from the impeller into the bulk fluid. Figure 8 shows the flow pattern of a mixer producing radial flow in two sections of the tank. In the bottom part of the tank, the impeller entrains the liquid in an upward direction and displaces it at right angles to the axis of the impeller. In the upper part of the tank the impeller entrains the liquid downward, also displacing it perpendicular to the impeller axis.

Axial flow, in which the liquid enters the impeller and discharges along a parallel path to the axis, is shown in Figure 9. The radial and longitudinal components are primarily responsible for the derived mixing action. The tangential component is important when the shaft has a vertical orientation and is positioned near the center of the tank.
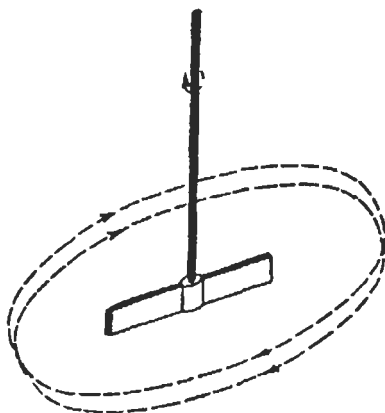
Figure 7. *Tangential flow pattern generated by a paddle mixer.*
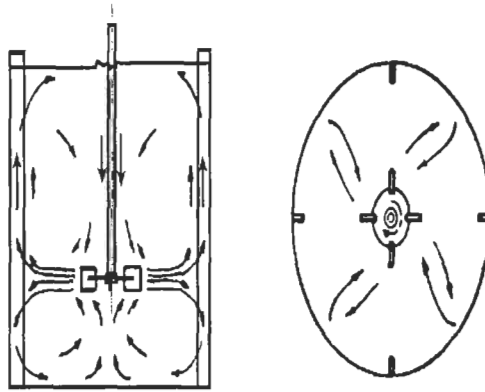
Figure 8. *Axial flow patterns in a tank.*
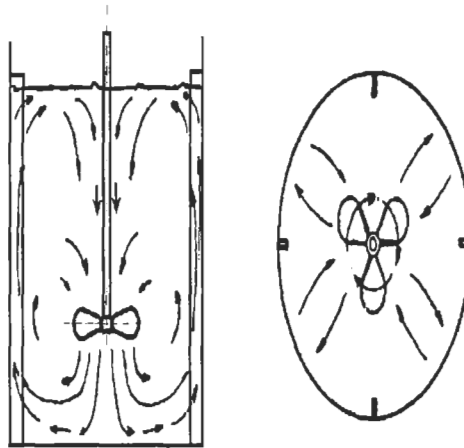
Figure 9. *Axial flow patterns.*

Based on the pitch of the impeller with regard to the direction of rotation, there are two possible axial flow patterns: that in which the impeller pumps the liquid from the bottom to the surface; and that in which the impeller pumps liquid from the surface to the bottom. A combination of the three principal types of flow normally is encountered in mixing tanks. The tangential flow following a circular path around the shaft forms a vortex at the surface of the liquid. The vortex formation results from the influence of gravity forces, quantitatively determined by means of the Froude number, which increases at higher speeds, promoting vortex formation. Figure 10 presents a three-dimensional flow pattern, affording a clear image of the liquid flow in the tank obtained by projecting the path of a liquid particle in two planes. Part (A) shows the path that the particle takes at a given impeller speed.
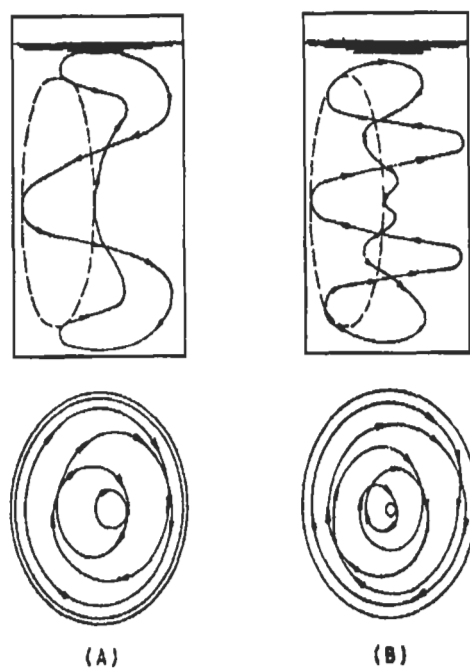
Figure 10. *Three-dimensional flow pattern.*

The particle undergoes four horizontal revolutions per single vertical revolution. As the impeller speed is increased, the particle performs a greater number of horizontal revolutions per one vertical revolution (Figure 10 (B)), showing that the greater centrifugal force at higher speeds increases the radius of curvature of the paths of the particle, which thus is shifted farther from the center of the tank. Further increases of the rotational speed and, subsequently, the centrifugal force, result in higher ratios between the horizontal and vertical revolutions of the particle and, thus, larger radii of curvature for particle paths. A particle subjected to gravitation also is acted on by tangential acceleration, as determined by the centrifugal force developed by the impeller, and influenced by the density and viscosity of the mixture. The direction of resultant acceleration is perpendicular to the surface of the liquid at any given point. If the tangential acceleration is negligible, the level of the fluid will be horizontal. Otherwise, the action of the centrifugal field becomes pronounced and the liquid level exhibits a depression that is referred to as the vortex.

Vortex formation is a condition that arises from centrifugal acceleration acting on gravitational acceleration. The circular motion of the entire contents of the tank predominates over the flow of the liquid from the impeller. Flow orientation thus is important not only in cases of noticeable vortex formation, but

also in mixers with tangential flow. In the latter case, at certain mixer dimensions and impeller speeds the circulating liquid in the tank may attain the same velocity as the impeller. Mixing becomes ineffective under such conditions.

Vortex formation leads to a considerable drop in mixing efficiency and should be suppressed as much as possible in practical applications to increase the homogenizing effects of mixers. The preferable method of vortex suppression is to install vertical baffles at the walls of the mixing tank. These impede rotational flow without interfering with the radial or longitudinal flow. Figure 11 illustrates such a system.

The distribution of velocity components (radial, tangential and axial) under conditions of mixing with baffles in comparison with the conditions of vortex formation is presented in Figure 12. The dashed lines in Figure 12 indicate non-baffled conditions. Comparison of the non-baffled and fully baffled velocity curves (solid line) leads to the following set of conclusions on vortex suppression when dealing with perfectly miscible liquids:

- Baffles are responsible for restricting the tangential velocity component, u, and augment the vertical component, while simultaneously increasing the radial velocity, U_r. The net result is that the liquid discharges from the impeller in a wider flow radius.
- The streamlined distribution becomes more regular when baffles are used. Hence, the energy transmitted from the impeller to the liquid is utilized more uniformly.
- The circulation increases, and the difference between the circulation rate under fully baffled conditions and at the vortex formation rises to two to four times the original value. This means that the power input increases considerably in the range of two to ten times the input without baffles.
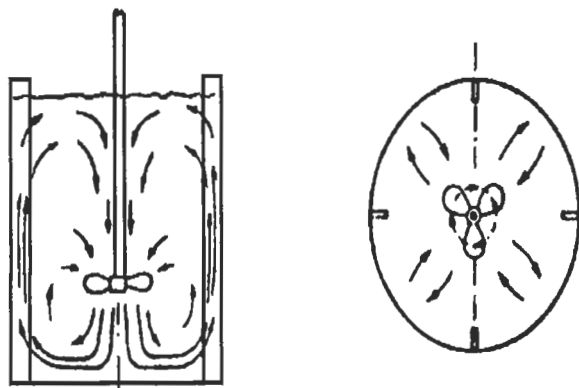
Figure 11. *Flow patterns in a baffled tank with centrally mounted impeller.*
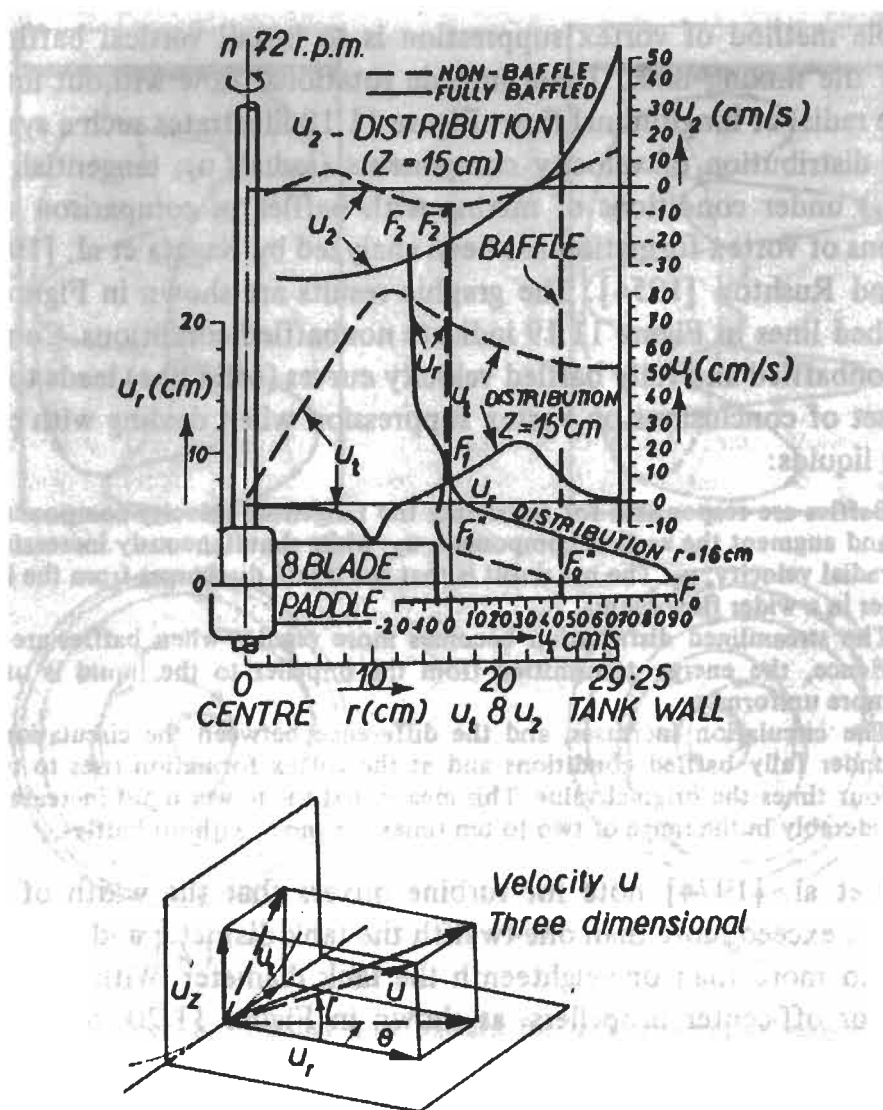
Figure 12. *Variation of velocity distribution in a mixing tank on insertion of full side wall baffles.*

For turbine mixers that the width of a baffle should not exceed more than one-twelfth of the tank diameter and, for propeller mixers, no more than one-eighteenth the tank diameter. With side-entering, inclined or off-center propellers, as shown in Figure 13, baffles are not required. Instead, shrouded impellers and diffuser rings may be used to suppress vortex formation. These devices contribute to flow resistance and reduce circulation by creating intense shear and abnormal turbulence

at the impeller discharge. The specific flow pattern in the tank depends on the type of impeller if vortex formation and swirling are eliminated. With propeller mixers, the liquid is driven straight down to the bottom of the tank, where the stream spreads radially in all directions toward the wall, flows upward along the wall, and returns to the suction of the propeller from the top. Paddle mixers provide good radial flow in the immediate plane of the impeller blades but are poor in developing vertical currents. Turbine mixers drive the liquid radially against the wall, where the stream divides into two portions. One portion flows downward to the bottom and back to the center of the impeller from below; the other flows upward toward the surface and back to the impeller from above. Figure 14 shows that there are two separate circulation currents generated.
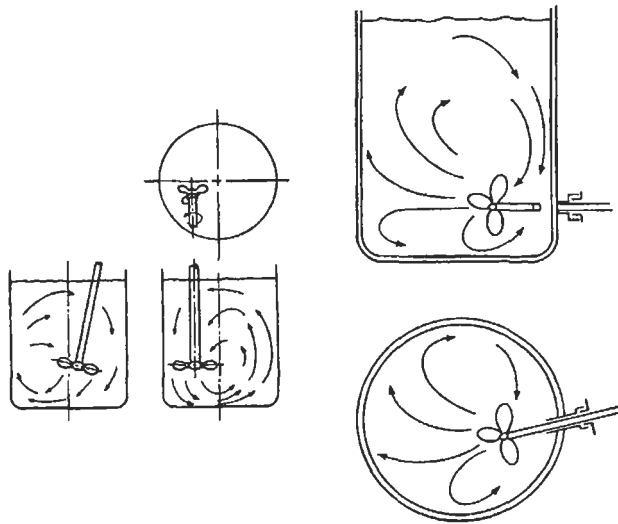
Figure 13. *Off-center impeller flow patterns.*
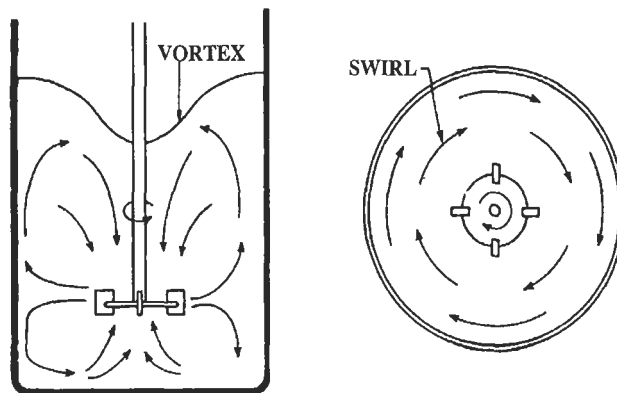
Figure 14. *Circulation patterns in an agitated tank.*
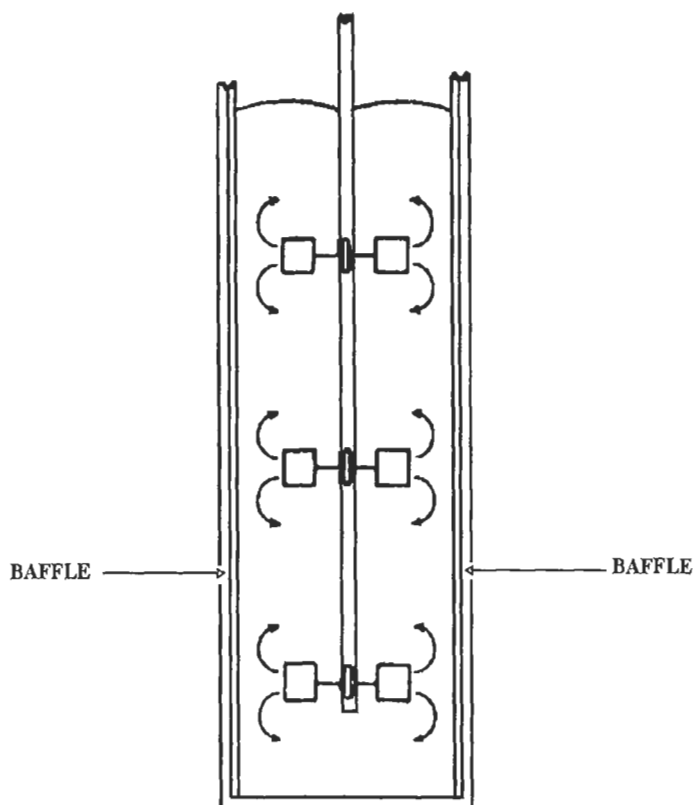
Figure 15. *Multiple turbines in a tank.*

Turbines are especially effective in developing radial currents, but they also induce vertical flows, especially under baffled conditions. In vertical cylindrical vessels, the ideal liquid depth for good mixing should be somewhat greater than the tank diameter. If greater depths are required, two or more impellers may be installed on the same shaft, with each impeller serving as a separate mixer. Two circulation currents are generated for each mixer, as shown in Figure 15 .

The bottom impeller should be mounted about one impeller diameter above the bottom of the tank. As noted earlier, when the direction and velocity of the flow to the impeller suction are to be controlled, draft tubes are employed. These devices are designed to set in motion regions in the tank that otherwise would not be agitated. Typical flow profiles are shown in Figure 16. Draft tubes are mostly used with propellers and really amount to mixing by circulation of the contents through a thoroughly agitated region in the vicinity of the impeller. The turbulence induced in the tank by the flow of liquid from the draft tube only contributes to the blending action.
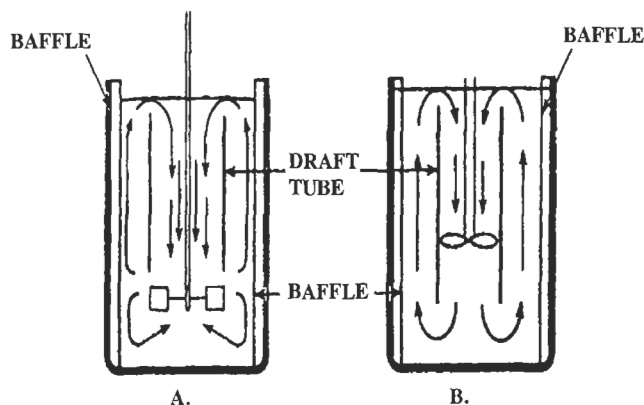
Figure 16. *Draft tubes in baffled tanks: (A) turbine; (B) propeller.*

DESIGN PRACTICES

In the previous section, a description of the various types of equipment commonly used for carrying out mixing operations was provided.   Emphasis was placed on mechanical agitators since these have the broadest range of application, however there are other important devices worth noting, such as jet, pump, eductor, orifice, and inline mixing devices. Some of these important pieces of equipment are briefly described below.

Specifying a mixing device is a two-stage process. Mixing needs of the application first have to be quantified and then these needs have to be translated to an equipment design. Included in this section is the translation from mixing need to specified equipment design. Principles addressing how to quantify the mixing need for systems of the common phase configurations of miscible fluids, gas-liquid, immiscible liquids, and solid-liquid are reviewed.

As noted earlier, mixing operations are carried out to blend two or more miscible fluids, to suspend solids in a liquid to form a slurry (which may then dissolve or react) , to disperse a gas into a liquid to carry out mass transfer, and to disperse one immiscible liquid into another for mass transfer or to form an emulsion. For any of these operations, equipment sizing and specification can be based upon a numerical description of the mixing requirement. This mixing equipment can be expressed as a given discharge flow rate from the mixing device, a given total mixing power or power per unit volume dissipation by the mixing device, a given bulk fluid velocity, or in the case of turbine agitators a given tip speed. Before addressing this in more detail, some basic terminology needs to be introduced:

Agitator: a general term used to describe a device which imparts motion to a fluid.

Agitator is a term most commonly used as a synonym for the more specific term impeller.

Baffle: generally a flat plate attached to and perpendicular to the wall of a mixing vessel to alter the fluid circulation pattern and prevent excessive swirl of the fluid in the vessel .

Critical speed: the mixer shaft speed which matches the first lateral natural frequency of the shaft and impeller system. Excessive vibrations and shaft deflections are present at this speed.

Driver: the motor and gear box combination used to rotate the impeller shaft.

Draft tube: a centrally located open ended tube in a mixing vessel which confines the impeller discharge or suction to produce a vertical flow in the vessel.

Geometric similarity: the sizing of the components of a mixing system in such a way that the ratio of linear dimensions of the components (impeller diameter, vessel diameter, vessel height, etc.) remains the same for differing overall system sizes.

Impeller: a physical device which rotates to impart motion to a fluid. Examples are turbines and propellers described earlier.

Impeller Reynolds Number: a dimensionless number used to characterize the flow regime of a mixing system and which is given by the relation $Re = \rho ND^2/\mu$; where ρ = fluid density, N = impeller rotational speed, D = impeller diameter, and μ = fluid viscosity. The flow is normally laminar for $Re < 10$, and turbulent for $Re > 3000$.

Jet: a fluid stream having a velocity greater than the surrounding fluid which discharges from a nozzle or impeller.

Mixer: a device for carrying out a mixing operation and which generally inclusively refers to an impeller, driver, baffle, and vessel combination.

Power Number: a dimensionless number used to describe the power dissipation of impeller and which is given by $N_P = Pg_c/\rho N^3D^5$; where P = impeller power dissipation and g_c = gravitational conversion factor.

Propeller Pitch: pitch is the advance of the fluid per revolution on the basis that a propeller is a segment of a screw. Normally "square" pitch is used; i.e., a pitch value equal to the propeller diameter.

Pumping Capacity: the amount of discharge flow from an impeller.   It is frequently correlated on the basis of the dimensionless impeller discharge coefficient, $N_Q = Q/ND^3$, where Q = volumetric discharge rate.

Side Entering: describes a mixer design which has a horizontal shaft entering through the side of a mixing vessel.

Standard Geometry: describes a vessel and mixer design based on a fluid depth equal to vessel diameter and a top-entering impeller having a diameter equal to 1/3 of vessel diameter and located with a clearance of 1/3 of vessel diameter above the bottom of the vessel.

Steady Bearing: a bearing located at the bottom of the shaft of a top entering impeller to minimize shaft deflection and vibration. It is immersed in the fluid being mixed.

Tip Speed: the peripheral speed of an impeller which is equal to πND, where N and D are defined as earlier..

Top Entering: describes a mixer design which has a vertical shaft entering through the top of a mixing vessel.

The commonly used types of mixing equipment can be placed in the broad categories: (1) mechanical agitators, (2) inline motionless mixers, (3) tank jet mixers, and (4) miscellaneous. The nature and type of agitator used depends upon the scale and type of mixing and upon the fluids being mixed. The broad classes of impellers are:

Turbine: Turbines are the most versatile and commonly used mechanical agitator. The most common turbine types are flat blade, disk , and pitched blade. Turbines are normally mounted on a vertical shaft. The turbine types differ in their discharge flow direction and thus the tank circulation pattern set up and in the way the mixing energy dissipation is divided between shear and flow. Flat blade and disk turbines have a radial discharge flow and tank circulation pattern. Both of these turbine types are considered to have a large portion of their energy dissipation in shear and are used in applications needing this characteristic such as chemical reaction and gas and liquid dispersion. The disk turbine is especially suited for gas dispersion applications since the disk prevents gas sparged beneath the turbine from bypassing the high shear discharge region. The blades of flat blade turbines can be either hub or strut mounted. Pitched blade turbines have a primary axial discharge flow which is normally directed downward. The pitched blade turbine is especially suited for applications requiring flow such as blending and solids suspension. Standard flat and pitched blade turbines can be modified to take a variety of forms. The blades can be curved, shrouds can be added to the top or bottom to control inflow, the blade areas can be concentrated at the tips, and stabilizing rings or blade projections can be added. References noted at the end of this chapter should be consulted for additional information concerning these special turbines.

Propeller : As noted previously, the discharge from a propeller is primarily axial. Propellers are the most efficient impeller for producing flow and are especially suited for blending operations. In addition to the top entering configuration, a side entering configuration is used for propeller agitators in large blending and storage tanks.

Paddle: A paddle is similar to a turbine impeller but typically has only two large blades and operates at lower speeds than a turbine. They are primarily used in high viscosity mixing operations. In European and Japanese literature the term "paddle" also is used to describe the flat blade and pitched blade turbines discussed above. The term "turbine" generally is reserved for disk turbines.

Close Proximity: Close proximity impellers are designed to have minimal clearance to the wall of the mixing vessel and are used for high viscosity mixing and agitated tank heat transfer applications.   Example types are gates, anchors, and helical impellers. The Uhl and Gray and Nagata references should be consulted for information concerning these impellers.

High Shear: High shear impellers take a variety of proprietary forms and are used primarily for producing emulsions. Their design maximizes the portion of the mixing energy dissipation which is classified as shear. High shear impellers are available for both tank and inline applications.

Inline motionless mixers derive the fluid motion or energy dissipation needed for mixing from the flowing fluid itself. These mixers include:

Orifice Mixing Column: An orifice mixing column consists of a series of orifice plates contained in a pipe. The pipe normally is fabricated of two vertical legs connected by a return bend at the bottom with the orifice plates installed between flanges in the vertical legs. Typical use is for cocurrent contacting in caustic and water washing operations.

Mixing Valve: This type of mixing device is normally a manually operated globe valve operated at 20 to 350 kPa pressure drop. Common use of this device in refinery operations is for water and crude oil mixing before a desalter and in caustic scrubbing operations.

As an alternate to mechanical agitators, storage and blending tanks can be agitated by *jet mixers*. Jet mixers can be used for continuously blending miscible liquids as they enter a tank or batchwise by recirculating a portion of the tank contents through the jet. They are commonly used to prevent stratification in product and intermediate tankage. The jet mixer is a streamlined nozzle installed in the side of a tank near the vessel bottom. It points diametrically across the tank and discharges at an angle above the horizontal. A typical jet nozzle design is shown in Figure 17. It is important to note that mixing is sometimes achieved as an adjunct of pumping or recirculation operations. In particular, centrifugal pumps and jet eductors provide short contact time mixing within the devices. A typical jet eductor is shown in Figure 18.
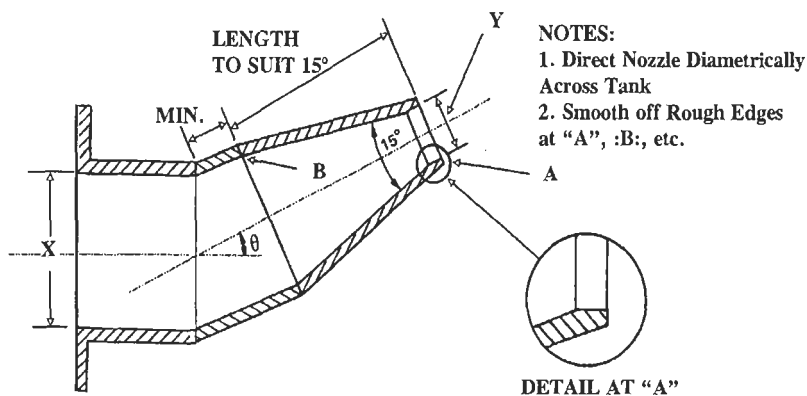
Figure 17. *Jet nozzle for tank mixing application.*
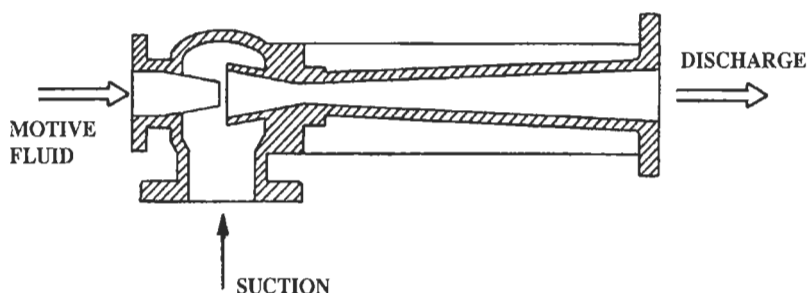
Figure 18. *Liquid jet eductor.*

The steps in mixing equipment design include: (1) identification of mixing need and quantification of requirements; (2) selection of general equipment type; (3) selection of equipment geometry; and (4) calculation of equipment specifications. Among the types of equipment described, questions involving general equipment type selection arise in connection with blending and storage tanks and in connection with inline mixers. For a blending or storage tank, the choice is between using a side entering propeller agitator or using a tank jet mixer. If the tank is filled by means of a pumping system having extra pressure drop capability or is equipped with a pump around recirculation system, a jet mixer should be considered. The choice among inline mixers is based on experience in a particular service, pressure drop considerations, and equipment availability. With any inline mixer, only radial mixing is achieved. The mixers cannot smooth out time variations in fluid properties. The remainder of this discussion is specific to mechanical agitators. In this regard, tank geometry is an important consideration for mechanical agitators. For large (> 4 m diameter) storage and blending tanks, a side entering propeller agitator is normally used. The agitator shaft is set at an angle to a tank diameter. No tank baffles are used. For very large (> 40 m diameter) storage tanks having high mixing power requirements, multiple mixers can be used. As noted earlier, these may be mounted on a single shaft, or multiple shafts.

Other mechanically agitated mixing operations normally use vertical mixer shafts in vessels equipped with wall baffles. However, shafts are inclined approximately 10° from the vertical in unbaffled vessels which employ propeller agitators for light duty applications. For permanent installations, vertical shafts on the tank centerline and wall baffles are recommended. Unless process requirements dictate otherwise, "standard" tank geometry is used. This standard geometry is illustrated in Figure 19. Standard geometry utilizes only one impeller on the mixer shaft. If an application such as gas absorption requires that the fluid depth, Z, be greater than 1.2 times tank diameter, T, an additional agitator spaced at least an agitator diameter, D, above the standard bottom agitator should be used. Standard geometry normally implies a top entering shaft.
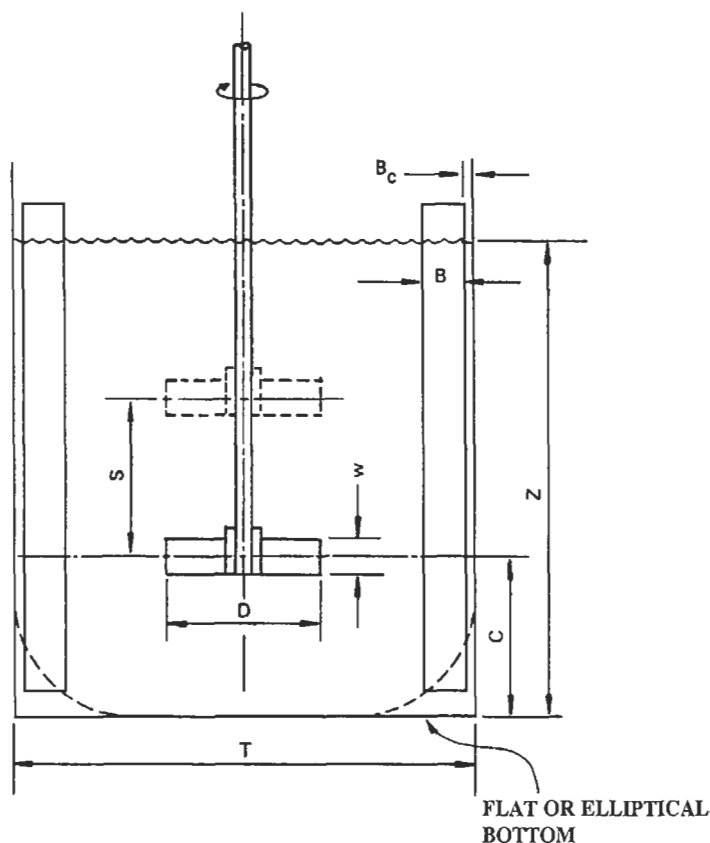
Figure 19. *Standard geometry tank.*

From a flow pattern and power dissipation standpoint, a bottom entering shaft is equivalent. If shaft seal problems are handleable, bottom entry can reduce the required shaft length in vessels which are designed with tall vapor spaces for foam disengagement, etc. The geometric parameters for a standard geometry tank shown in Figure 19 are defined as follows: $Z = T$, $D = T/3$, $B = T/12$ to $T/10$, $B_c = T/12$, $w = D/8$ to $D/5$.

Power Dissipation and Discharge Flow Correlations

The power dissipation and discharge flow of the various types of mechanical agitators are correlated on the basis of a dimensionless power number, N_P, and dimensionless discharge coefficient, N_Q, both of which are functions of the impeller Reynolds number, Re. For common turbine agitators, power numbers are correlated in Figure 20 for different agitator types. For standard 45° pitched blade turbine agitators, discharge coefficients based on total established flow are given in Figure 21. For propeller agitators, the power numbers are given in Figure 22. For

propeller agitators operating at turbulent Reynolds numbers (Re > 3000), the direct discharge flow can be calculated from $N_Q = 0.5$. The power dissipation of a mixing impeller is given by the following correlation:

$$P = N_P \, \rho N^3 D^5 / 10^3$$

where: P = power dissipation, kW

N_P = power number, dimensionless (see Figure 20 or 21)

ρ = fluid density at conditions, kg/m^3

N = impeller rotational speed, rev/s

D = impeller diameter, m

Since N_P is practically constant under turbulent operating conditions (Re > 3000), power is proportional to the 3rd power of impeller speed and the 5th power of impeller diameter. The power numbers given in Figures 20 and 21 apply to the impeller types indicated and specifically when these impellers are employed in a tank of standard geometry which includes wall baffles (except Curve 7 of Figure 20 specifically applies for the finger baffles shown). For side entering propeller agitators which are offset from or set at an angle to a tank diameter, Figure 22 is still applicable. For mixing liquids having viscosities greater than 5 Pa-s, baffles can also be eliminated in other vessels without altering the power number relationship. Impeller power dissipation can be estimated for a number of departures from the standard geometries. For turbine blade width/diameter ratios other than shown in Figure 20, the power number can be calculated as being proportional to this ratio.
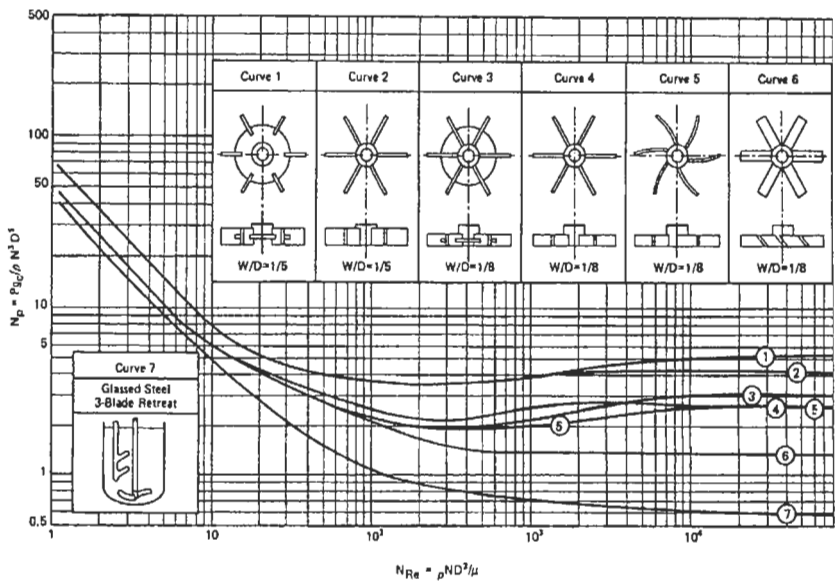
Figure 20. *Correlations for mixing power number vs. Reynolds number.*
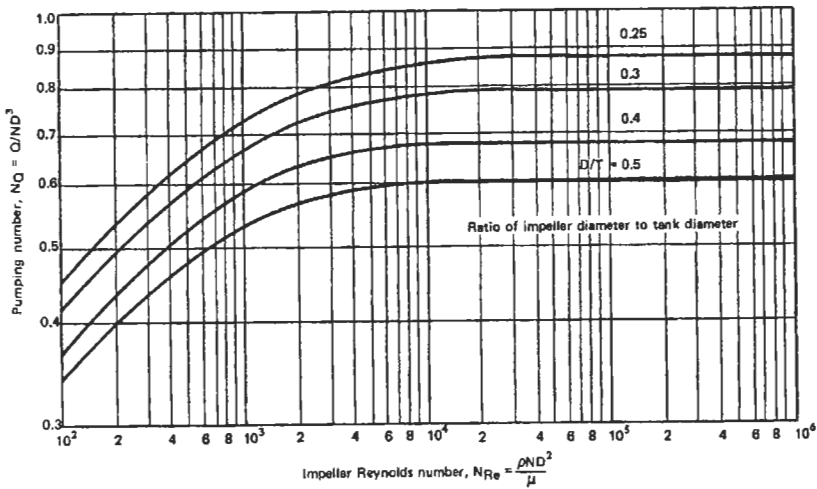
Figure 21. *Discharge coefficient for 45° pitched blade turbine vs. impeller Reynolds number.*
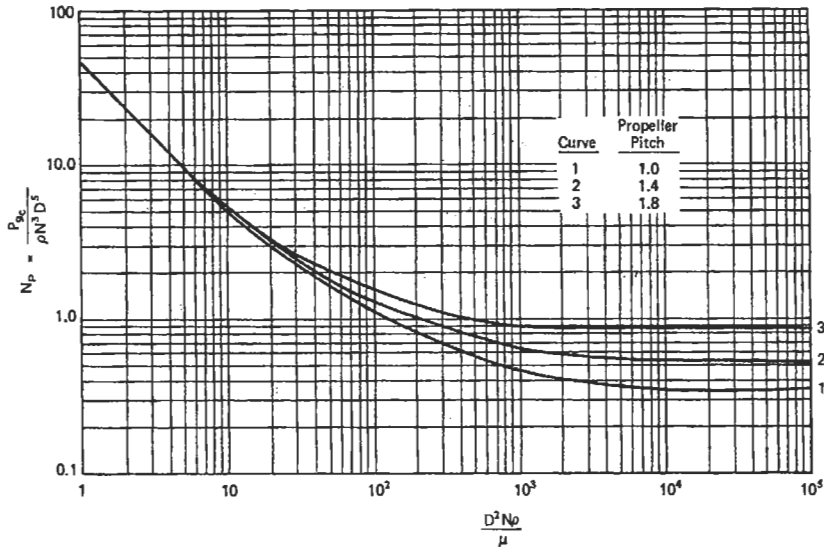
Figure 22. *Propeller power number correlations.*

For installations having multiple impellers on the same shaft, the power dissipation is a function of impeller spacing as shown in Figure 23 for turbines and Figure 24 for propellers. Changes in baffle width from the standard T/12 to T/10 affect power number as shown in Figure 25. The sparging of a gas into a mixing vessel reduces power dissipation as indicated in Figure 26. Figures 23 through 26 give the ratio

of power number for the non-standard geometry (or gas sparging situation) to the power number for standard geometry (or no gas flow). Thus, the power number for the non-standard situation is the product of the appropriate standard value times the ratios from the figures.
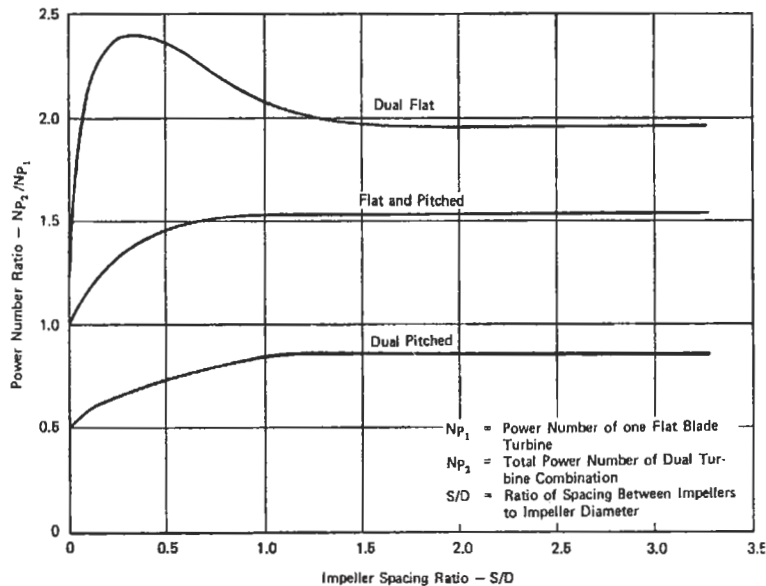
Figure 23. *Plot shows effect of dual turbine spacing on power.*
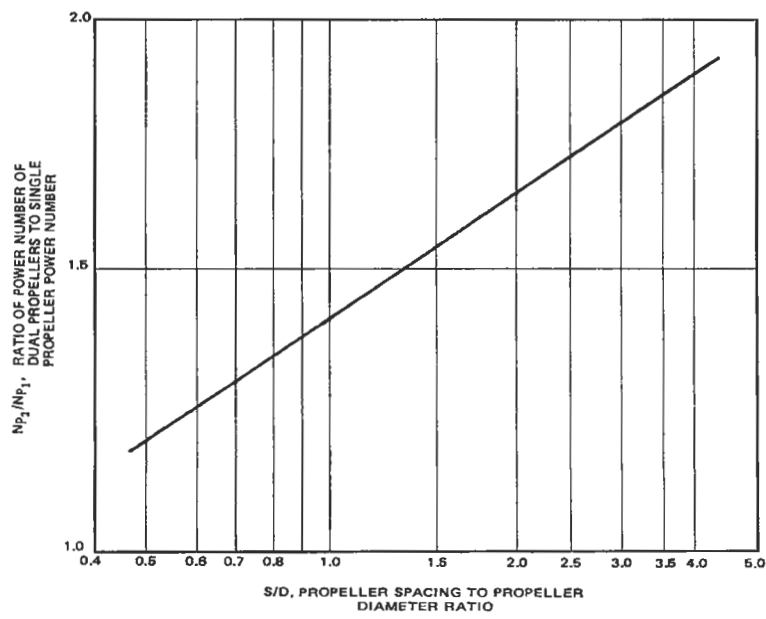
Figure 24. *Effect of dual propeller power.*
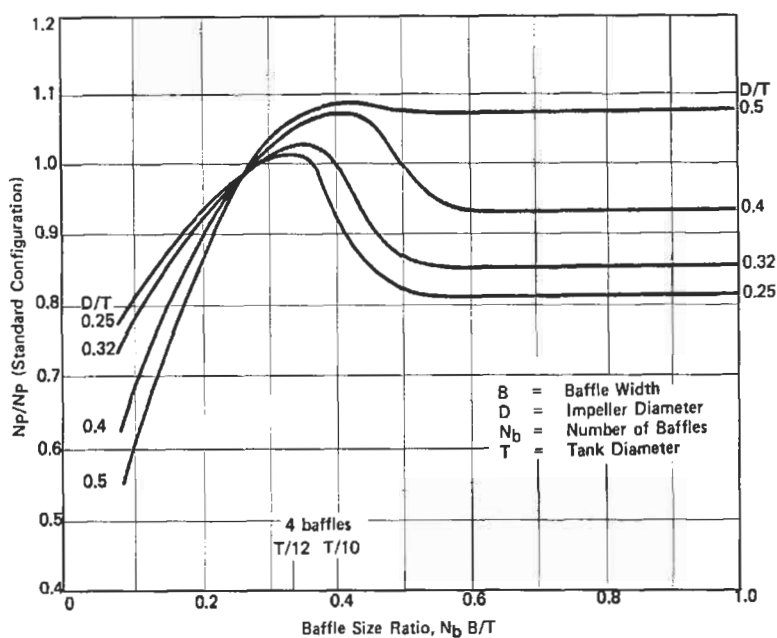
Figure 25. *Plot provides effect of baffling and D/T on the power number.*
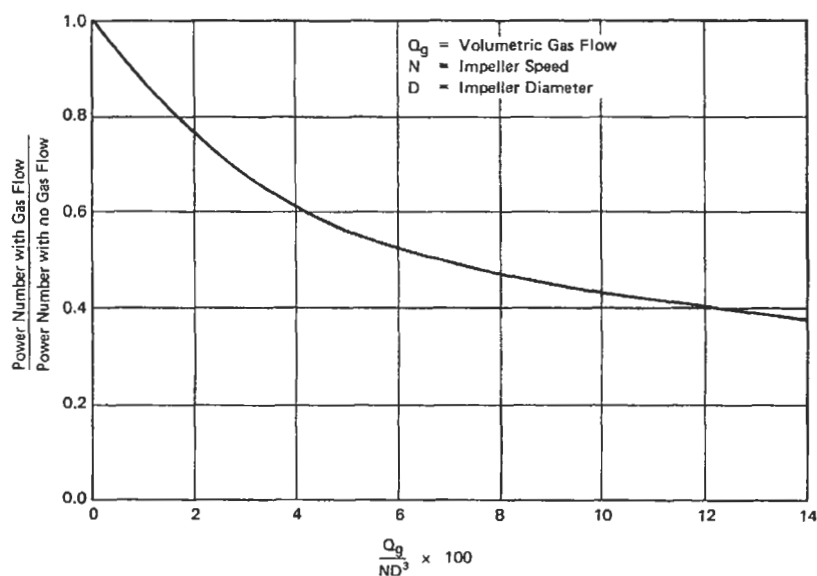
Figure 26. *Plot gives effect of gas addition on power.*

The power rating of the driver for the impeller has to exceed the power dissipation to the fluid because of driver, seal, and bearing losses. Unless specific data are

available, these losses can be estimated as 25% of the calculated dissipation. This 25% also provides a safety factor for uncertainties in the correlations and fluid properties used in calculating the dissipation to the fluid.

The power dissipation correlations discussed are specifically applicable to the mixing of fluids that are Newtonian. A Newtonian fluid has a viscosity independent of the shear to which the fluid is subjected. Thus, although the shear rates vary greatly throughout the fluid in an agitated vessel, the viscosity of a Newtonian fluid will be the same at all points in the vessel. In contrast, the apparent viscosity of a non-Newtonian fluid at any point in the vessel depends on the magnitude of either the shear stress or the shear rate at that point and may also depend upon the previous history of the fluid. If the apparent viscosity decreases with increasing shear rate, the fluid is called pseudoplastic. If the apparent viscosity increases with shear rate, the fluid is termed dilatant If the apparent viscosity of a pseudoplastic fluid decreases with the time a particular rate of shear is applied, the fluid is termed thixotropic. The Newtonian/non-Newtonian behavior of a fluid or fluid mixture is known only from prior experience or by viscometry. Many non-Newtonian fluids encountered are pseudoplastic. For these fluids studies have established that the power number versus impeller Reynolds number behavior is such that the power number is always equal to or less than the value for a Newtonian fluid. Thus, for pseudoplastic fluids, Figures 20 and 22 can still be used and will provide conservative numbers. If the agitation conditions are known to be turbulent and result in operation on the flat portion of the power number-Reynolds number curve, a precise knowledge of apparent viscosity is not needed. However, in the laminar range, a knowledge of average shear and thus apparent viscosity is needed to calculate the appropriate Reynolds number. In a stirred tank the average shear rate is equal to approximately 10 times the rotational speed of the impeller. Experimental measurements are required to determine apparent viscosities at this shear rate.

We now focus attention on mechanical agitation in a fixed vessel geometry. For large storage and blending tanks which use side entering propeller type agitators, the vessel geometry is established by mechanical and economic rather than mixing considerations. In other words, the mixer design is adapted to a tank of specified geometry. For vessels to be mixed with top entering agitators, "standard geometry" (Figure 19) should normally be used. Vessel diameter, T, can be estimated from the following relationship:

$$T = [4V/\pi]^{1/3}$$

where V is the maximum fluid volume to be mixed in the vessel. This diameter is then rounded to the nearest larger available drum and head diameter (typically available in 100 or 200 mm intervals). Liquid depth in the vessel can then be calculated. Depth and volume in the bottom head are included. The head can be any of the standard types available for the pressure rating of the vessel. Vessel volume

above the fluid must be added in many installations to provide for the increase in liquid volume due to gas holdup during sparging or to accommodate foaming. In all cases space is left between the top of the liquid and the top of the vessel, thus the liquid has a free interface. Normally for vessels with top dished heads, the space above the upper tangent line is left unfilled. With flat heads or open tops, the vertical space left unfilled should be at least 1/10 of vessel diameter. For mixing fluids having viscosities less than 5 Pa-s, 4 full length wall baffles spaced 90° apart are used. In vessels having dished heads, the baffles are only used on the straight side. Except when mixing buoyant particle slurries, baffle width is set at 1/10 to 1/12 of vessel diameter. For buoyant solids, baffle width is reduced to 1/50 of vessel diameter. When solids are present, the baffles are set out from the vessel wall by 1/72 of vessel diameter. If the fluids in the vessel will always have a viscosity greater than 5 Pa-s, wall baffles are not needed. For mixing fluids having viscosities up to 1 Pa-s, impeller diameter is set at approximately 1/3of vessel diameter. As fluid viscosity increases to 10 Pa-s, impeller diameter should be increased to approximately ½ of vessel diameter. Except for close proximity agitators, agitator diameter should not exceed 0.6 times vessel diameter for any viscosity. Except for very small impellers, impeller diameters are specified in even 10 mm increments.

For the set vessel geometry and approximate impeller diameter, impeller speed is then calculated to satisfy process requirements. For process requirements as stated as a tip speed, impeller speed is given by the following relationship:

$$N = \phi/\pi D$$

where ϕ is the tip speed. The dimensions used in this equation are m/s for tip speed, m for the impeller diameter, D, and rev/s for impeller speed N.

For a process requirement stated as a power unit per unit volume, impeller speed can be computed from:

$$N = [10^3 (P/V) (V)/N_p \rho D^5]^{1/3}$$

where N_p is the dimensionless power number, P/V is the power per unit volume (kW/m^3), V is the vessel volume (m^3), and is the fluid or slurry density (kg/m^3). In order to use Figure 20 or 22 to obtain N_p values, information on the impeller Reynolds number is needed. This can be calculated from:

$$Re = \rho ND^2/\mu$$

where μ is the fluid or slurry viscosity in units of Pa-s. Since N must be known to calculate the Reynolds number, an iterative calculation using the above equations may be required. However, in the turbulent regime, N_p is constant and independent of Re. For non-standard geometries and for gas sparging, the power number must

be additionally modified as given in Figures 23 through 26. For a process requirement stated as an impeller pumping capacity or discharge flow, impeller speed is given by the following formula:

$$N = Q/(N_Q D^3)$$

where: N_Q = impeller discharge coefficient, dimensionless
         Q = pumping capacity or discharge flow, m^3/s

The impeller discharge coefficient, N_Q, for pitched blade turbines on the basis of total flow is given in Figure 21 as a function of impeller Reynolds number. Reynolds number is calculated by the earlier expression and in other than the turbulent region may require an iterative calculation. For "square pitch" propellers operating in the turbulent region, a value of 0.5 can be used for N_Q. This value is based on direct discharge flow. For the selected vessel geometry and a selected impeller type and calculated impeller speed and diameter, the power dissipation to the fluid can be estimated from:

$$P = 10^{-3}[N_P \rho N^3 D^5]$$

where P is the power dissipation in units of kW. The power number, N_p for the specific mixing situation should be calculated as indicated earlier. In the case of vessels which have multiple functions or whose contents have varying properties, the maximum expected value of N_p should be used. To allow for seal and gear reducer losses and correlation and physical property uncertainties, the calculated power dissipation is multiplied by 1.25 to obtain the driver power. If the impeller could be covered by settled solids before being turned on in a solids suspension application, additional correction is required. For example in phosphoric acid attack tanks, an overall multiplicative factor of 1.75 is recommended. Specification of an agitated tank can be approached from an equipment specification or from a duty specification viewpoint. Equipment specification typically includes vessel dimensions; nozzle or sparger description and locations; impeller type, size, rotational speed or range of speeds, and position in vessel; baffle configuration; shaft and bearing description; driver power rating and service factor; and materials of construction. With axial flow impellers the direction of fluid flow must be indicated. Additionally with pitched blade turbines, blade width must be identified as being the actual width or as the projected width (height) parallel to the axis of rotation of the turbine. The following sample calculations illustrate the use of some of the formulas presented above.

Sample Calculation of Mechanical Agitator Design

Problem: A mixer is needed for a 2 m^3 blending tank containing fluids having a density of 1,000 kg/m^3 and a viscosity of 0.01 Pa-s. The process requirement is that

the mixer have a circulation capacity of 0.2 m³/s. Determine the appropriate tank geometry, mixer type and size.

Solution: "Standard geometry" is appropriate for this application (see Figure 19). The vessel volume is 2 m³. The trial vessel diameter is computed as follows:

$T = [4V/\pi]^{1/3} = [4 \times 2 \div \pi]^{1/3} = 1.37$

We round this off to T = 1370 mm, which for a flat bottom tank is Z = 1360 mm. The vessel should have four full length wall baffles having a width between T/12 and T/10. Thus B = 130 mm.

A top entering pitched blade turbine having w/D = 1/8 is appropriate for this blending service.

A trial turbine diameter: D = T/'3 = 457 mm.

Process requirement is stated as a pumping or circulation capacity of 0.2 m³/s Thus trial turbine speed is computed as follows: $N = Q/(N_Q D^3) = 0.2/\{(0.75)(0.457)^3\}$ = 2.79 rev/s. The value of $N_Q = 0.75$ is from Figure 21 for $Re > 10^4$. The calculated speed of 2.79 rev/s should be rounded to the standard gear driver output speed of 2.58 rev/s, based on vendor recommendations. To more closely satisfy the process requirement after shaft speed rounding, turbine diameter D can be recalculated from $D = [Q/N_Q N]^{1/3} = [0.2 / (0.75) (2.58)]^{1/3} = 0.469$ m, which after rounding is 470 mm. The Reynolds number must be calculated to check the validity of N_Q from Figure 21. Thus, $Re = (1,000)(2.58)(0.470)^2/0.01 = 5.7 \times 10^4$, which indicates operation is in the turbulent regime as assumed.

The power dissipation to the fluid is then calculated as: $P = 10^{-3} N_P \rho N^3 D^5 = (10^{-3})$ (1.3) (1,000) (2.58)³ (0.470)⁵ = 0.51 kW. The value of $N_P = 1.3$ is from Figure 20 for a pitched blade turbine of w/D = 1/8. Driver power (which includes mechanical losses) is estimated by multiplying this dissipation by 1.25 to obtain 0.64 kW. This power is rounded up to a standard electric motor rating of 0.75 kW.

To summarize, the design would be: T = 1370 mm, Z = 1360 mm, B = 130 mm, C = 460 m (from Figure 19), D = 470 mm, w/D = 1/8, N = 2.58 rev/s, and P = 0. 75 kW.

Mixing of Miscible Liquids

The mixing of miscible fluids is commonly practiced. This mixing can be for blending components having different physical properties or can be for ensuring the homogeneity of a single fluid. For the blending or mixing of miscible fluids a criteria describing the desired approach to homogeneity must be set. Examples of such criteria are maximum allowable deviation of a fluid property (i.e., concentration, conductivity, color, temperature, etc.) at a point from the average value at that point, maximum allowable deviation of a fluid property between two or more points, etc. In addition to homogeneity criteria, blending operations can be specified on the basis of an empirical batch mixing time, ratio of mixer discharge flow to stream flow rate (or for batch systems, tank volume divided by batch time),

or a mixing power per unit volume to be supplied to the contents of the mixing vessel. Miscible fluids can be blended in inline mixing devices or in vessels. The inline devices are used when the components of the mixture are fed in the exactly desired proportion to the mixing device on a continuous basis. The inline device then removes any radial variations across the line cross-section. Inline devices do not provide back mixing; hence they cannot be used to smooth out fluid properties which vary with time. The inline mixers are particularly useful upstream of analyzer sample points in computer controlled blending systems. Vessel mixing of miscible fluids can be done on a batch, semi-batch, or continuous basis. In a batch operation, all components are present before the mixing starts. In a semi-batch operation, mixing is underway as the components are added. The components can be added together or sequentially. For vessel mixing, three distinct configurations are generally used: jet mixers, side entering propellers, and top entering propellers and turbines. Jet mixers are used in conjunction with a pump which serves as the source of the required mixing energy. If the supply pumps have sufficient ΔP capability, tank jet mixers are attractive for use in LNG, LPG, gasoline, jet, and distillate fuel storage and blending tanks. Side entering propeller mixers are used in blending and storage tanks from 4 to 90 m in diameter. Side entering propellers are the normal mixing device for crude oil storage tanks. Top entering mixers are used for small tanks and for tanks which have high mixing requirements such as reactors and flash mixing tanks used in wastewater treatment.

The basic requirements to be satisfied by the mixer are that the entire contents of the tank be mixed and that the mixing be completed within the desired time period. Consideration of the entire contents arises because if fluid batches of different densities are added to a tank, it is possible to form stable layers of the different batches. This is known as stratification. In a stratified tank, mixing accomplished by a mixer physically located in one layer does not guarantee mixing throughout the entire tank. The differing fluid densities which lead to stratification can be caused by differences in temperature or composition. In the case of LNG and LPG, which can vary in composition, it is possible to add a dense layer to the bottom of a tank which upon warm-up during storage can become less dense than the upper layer resulting in a "roll-over" of the tank contents. This can result in dangerously high vapor release rates due to spontaneous flashing of the warm layer which is suddenly exposed to reduced pressure at the surface of the tank. For operations where stratification can potentially occur, tank operating procedures and mixer design should be selected so that stratification will not occur. Once a tank becomes stratified, unusually high mixing energy is required to break the stratification.

Jet mixers can be used for continuously blending miscible fluids as they enter a tank or batchwise by recirculating a portion of the tank contents through the jet. The jet mixer is a stream-lined nozzle installed in the side of' a tank near the bottom. It points diametrically across the tank and discharges at an angle above the horizontal. Jets accomplish mixing by the entrainment of the surrounding fluid into the jet. The induced flow within the tank is therefore greater than the jet flow itself and can lead

to rapid mixing. The amount of fluid entrained by a jet is a function of jet Reynolds number, jet expansion angle, and jet length. Turbulent jets, i.e., jet Reynolds number >3000, provide the most effective mixing.

Entrainment is dependent on jet expansion angle and length. Expansion angle is a function of the kinematic viscosity of the jet fluid as given by:

$$\tan \delta = 0.810 \, \upsilon^{0.133}$$

where δ = jet half angle, angle between edge of expanding jet discharge and jet centerline degrees, and υ = kinematic viscosity, m^2/s.

Since few experimental data are available for liquids having kinematic viscosities greater than $7 \times 10^{-6} \, m^2/s$ for conservative design, values for δ should be limited to that corresponding to $7 \times 10^{-9} \, m^2/s$ even for more viscous liquids. The total flow produced by a free jet of circular cross-section (the type produced by a tank jet mixer) is given by:

$$W_x = 2 \tan(\delta) \, (x/d) \, W_o, \text{ for } 5 < x/d < 120$$

where: W_x = total mass flow at distance, x, kg/s
  W_o = mass flow discharge from nozzle, kg/s
  x = downstream distance from nozzle, m
  d = nozzle exit diameter, m

The velocity of the jet decreases with increasing distance from the nozzle. The axial velocity along the jet centerline (the location of the maximum) is given by the following empirical expression:

$$U_x = 5(d/x)U_o, \text{ for } 5 < x/d < 120$$

where: U_x = centerline velocity at distance, x, m/s
  U_o = velocity at nozzle exit, m/s

The jet centerline velocity is used to estimate the jet penetration distance above the surface at low tank liquid levels or to calculate forces on floating roof structures. The restriction of the above equations to downstream distances (x/d) of less than 120 is due to generally low velocities and lack of data beyond that point. The following outlines a design procedure and formulas for jet mixing. The design parameters of interest are the Angle of Inclination, Nozzle Discharge Velocity, Nozzle Diameter, Nozzle Pressure Drop, and Tank Mixing Time. Each is noted below.

Angle of Inclination, θ: The projection of the nozzle centerline should strike the liquid surface at maximum tank level at a point approximately 2/3 of the way toward the opposite side of the tank. The system geometry is illustrated in Figure 27. The principle formula is as follows:

$$\theta = \arctan (3H_S/2T).$$

where: θ = nozzle angle of elevation above horizontal, degrees, H_S = submergence of nozzle below liquid surface, m, T = tank diameter, m.

Nozzle Discharge Velocity, U_o: Nozzle discharge velocity is critical when the fluid discharging in the jet is of higher density than the contents of the tank. When the jet fluid is less dense than the tank contents, the following criteria are not controlling. First, the required nozzle discharge velocity for heavy into light fluid is given by:

$$U_o = [2gFH_S/\sin^2(\theta + 5^\circ) \times (\Delta\rho/\rho_o)]^{1/2}$$

where: U_o = nozzle discharge velocity, m/s
  g = acceleration of gravity, m/s^2
  F = dimensionless parameter depending on limiting conditions noted below
  $\Delta\rho$ = density difference between jet liquid and tank contents, kg/m^3
  ρ_o = jet fluid density, kg/m^3

Dimensionless parameter F can be computed accordingly:
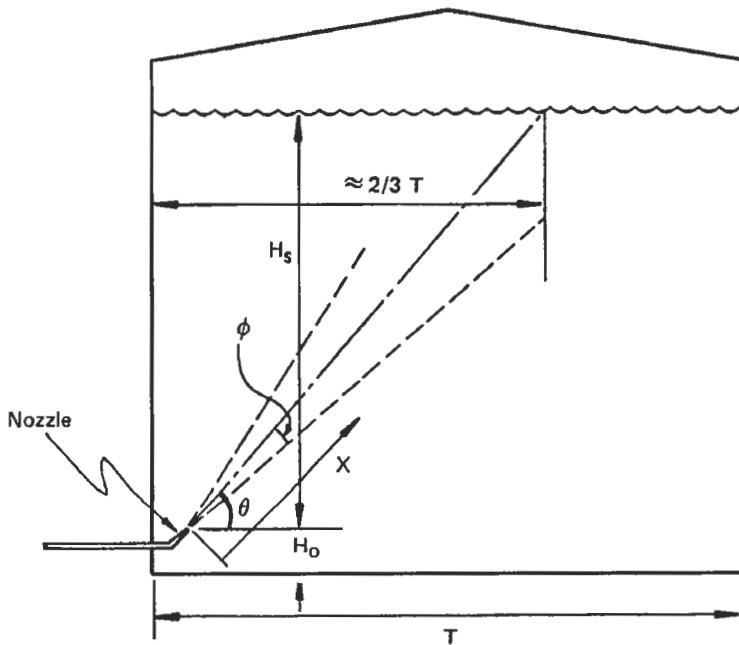
Figure 27. *Tank jet nozzle installation.*

To prevent stratification in an initially homogeneous tank:

$$F = 0.253 \, (\Delta\rho/\rho_o)^{-0.342} \, (H_S/d)^{0.652} \text{ for } H_S/d \leq 100, \text{ or}$$

$$F = 7.5 \, (\Delta\rho/\rho_o)^{-0.237} \text{ for } H_S/d > 100.$$

Where d is the nozzle exit diameter, m.
To destratify an already stratified tank:

$$F = 0.586 \, (H_S/d) - 4 \, \ell n(\Delta\rho/\rho_o) - 32.7 \text{ for } H_S/d > 50.$$

To prevent stratification in an initially homogeneous tank which is known to be already in jet induced circulation, assume 0.5F of the values computed from the above expressions.

Normally jet mixing nozzle design should be based on the first case above. The limiting design conditions (maximum required U_o) depends upon the way the tank is operated since the value of ΔP to use is that prevailing when the tank is filled to the level under consideration. Once filling is underway, the density difference declines as the tank fills due to the blending of the light and heavy liquids. Large heels may require impractically high nozzle exit velocities with heavy jet liquids. Lower velocities are possible if the heavy liquid is diluted with recirculated tank liquid before being added to the tank through the jet nozzle.

Nozzle Diameter, d: Nozzle exit diameter will be equal to or less than the diameter of the line feeding the tank. For a known flow rate of fluid supplied to the jet, the diameter is set by the largest size that will satisfy the requirement that the jet be turbulent or will satisfy the nozzle discharge velocity requirement (if the jet is denser than the tank liquid). For a turbulent flow requirement (both heavy and light jets):

$$d \leq Wo/750\pi\mu = Qo/750\upsilon$$

where: W_o = mass flow rate of jet fluid, kg/s
μ = viscosity of jet fluid, Pa-s
Q_o = volumetric flow rate of jet fluid, m³/s
υ = kinematic viscosity of jet fluid, m²/s

or, based upon a velocity requirement (heavy jets only):

$$d \leq [(4/\pi) \, (W_o/\rho_o U_o)]^{1/4} = [4Q_o/\pi U_o]^{1/2}$$

Additionally for adequate penetration of the nozzle discharge across the tank, the exit diameter should be at least as large as that given by (for both heavy and light jets):

$$d \geq H_S \,/\, 120 \, \sin \theta$$

Nozzle Pressure Drop, ΔP: The pressure drop through a jet mixing nozzle is given by:

$$\Delta P = 8.98 \times 10^{-4} \, W_o^2/\rho_o d^4 = 8.98 \times 10^{-4} \, \rho_o Q_o^2/d^4$$

where: ΔP = nozzle pressure drop, kPa. In addition to the nozzle pressure drop, contraction losses, kinetic energy changes, line losses, and the static head of liquid in the tank must be included in overall pressure drop calculations.

Tank Mixing Time, τ: The mixing time for adequate blending can be calculated by consideration of the amount of tank fluid circulated by jet entrainment in relation to tank volume. The ratio of circulation during the blending time to tank volume is called the number of tank turnovers. For blending to practical homogeneity, 10 tank turnovers are recommended. For blending to an approximate 1 % deviation between top and bottom sample points in a tank, three tank turnovers are normally adequate. The blending time for 3 tank turnovers when using a jet mixer as designed above is given by:

$$\tau = 1.45 \, \{d\rho_o T^2 (H_S + H_o) \sin \theta\} \,/\, (W_o \, H_S \, v^{0.133}), \text{ or}$$

$$\tau = 1.45 \, \{dT^2 (H_S + H_o) \sin \theta\} \,/\, (Q_o \, H_S \, v^{0.133})$$

where: τ = required blending time, s
  H_o = elevation of nozzle discharge above tank floor, m
  Q_o = nozzle volumetric flow rate, m^3/s
  v = kinematic viscosity, m^2/s

If the calculated blending time is longer than desired, nozzle discharge flow rate can be increased or, nozzle diameter increased. Consideration of nozzle recirculation line pressure drops and pump characteristics is required to select the parameter to change.

In locating a jet nozzle in a tank, care must be taken to insure that the direct nozzle discharge will not be blocked by outlet vortex breakers, tank roof supports, tank heaters, or other obstructions. The suction for a recirculating jet mixing system normally is taken diametrically opposite the nozzle or in the vicinity of the nozzle itself. Several precautions in the use of jet nozzles must be observed. In non-blanketed tanks, electrostatic charge problems can occur at low tank levels if the jet stream breaks through the liquid surface and breaks into a spray or mist. To avoid this problem, a low flow rate through the nozzle or an alternate horizontal inlet should be used until the jet nozzle is covered by sufficient liquid. Generally, 2 meters is adequate coverage. In floating roof tanks equipped with high velocity jet nozzles, low flow rates or a horizontal inlet should also be used to avoid roof

damage at low tank levels. Calculations of jet velocities at the surface should be based on jet centerline velocity. The use of jet mixing may not be feasible in some floating roof tanks because of restrictions on the jet exit velocity.

Gas-Liquid Mixing

Gas-liquid contacting is achieved by dispersing the gas in the liquid to increase the interfacial area for mass transfer. The overall objective of the contacting is the accomplishment of a chemical reaction or a gas stripping or absorption operation. The efficient carrying out of a mass transfer operation is dependent upon establishing a large interface between the gas and liquid and then maintaining this interface long enough for the desired mass transfer to occur. The rate of mass transfer can be conveniently expressed in terms of:

$$r_A/V = k_L a \, \Delta C_A$$

where: r_A = rate of transfer of component A, kg/s or k mol/s

   V = volume of system, m^3

   k_L = liquid phase mass transfer coefficient, m/s

   a = interfacial area per unit volume, m^{-1}

   ΔC_A = $(C_{Ai} - C_A)$ concentration driving force (difference between concentration of component A at the liquid-gas interface and the bulk liquid), kg/m^3 or moles/m^3 .

This form is particularly appropriate when the gas is of low solubility in the liquid and "liquid film resistance" controls the rate of transfer. More complex forms which use an overall mass transfer coefficient which includes the effects of gas film resistance must be used otherwise. Also, if chemical reactions are involved, they are not rate limiting. The approach given here, however, illustrates the required calculation steps. The nature of the mixing or agitation primarily affects the interfacial area per unit volume, a. The liquid phase mass transfer coefficient, k_L, is primarily a function of the physical properties of the fluid. The interfacial area is determined by the size of the gas bubbles formed and how long they remain in the mixing vessel. The size of the bubbles is normally expressed in terms of their Sauter mean diameter, d_{SM}, which is defined below. How long the bubbles remain is expressed in terms of gas hold-up, H, the fraction of the total fluid volume (gas plus liquid) which is occupied by gas bubbles.

Any type of equipment which produces a large interfacial area between gas and liquid phases can be used for gas-liquid mixing, contacting, or reaction. In addition to mechanically agitated vessels, possible equipment types include inline mixers and bubble, packed, and sieve columns. Mechanically agitated vessels produce backmixed contacting. Staging using separate vessels is required if other contacting patterns are desired. The other equipment types mentioned can in certain arrangements provide cocurrent plug flow or countercurrent staged contacting.

Mechanically agitated vessels for gas-liquid mixing are commonly used in alkylation; Paramins, chlorobutyl, and neoacids production; and wastewater treatment. Arrangements based on inline mixers are also used in wastewater treatment. The calculation procedure presented in this subsection is directed toward the use of mechanical agitators. The same approach applies for other mixing devices. If the gas bubble sizes and interfacial areas generated by these other mixing devices are known, the mechanical agitation design procedure can be easily adapted for use. Mechanical mixers for gas-liquid contacting normally use flat blade disk turbine agitators and the calculation procedures in this subsection are based on this style of agitator. The gas to be dispersed is usually added to the vessel through a ring sparger located below the agitator. For even gas distribution between sparger holes, the number and size of holes should be selected so that the gas velocity through the holes is at least 3 times the velocity through the pipe that forms the sparger ring.

To apply the mass transfer equation for design, the interfacial area, a, and mass transfer coefficient k_L must be calculated. The interfacial area is dependent upon the bubble size and gas hold-up in the mixing vessel as given by:

$$a = 6H/d_{SM}$$

where: a = interfacial area per unit volume, m^{-1}
H = gas phase hold-up (volume of gas/volume of gas + liquid), dimensionless
d_{SM} = Sauter mean bubble diameter, m

The hold-up and bubble diameter in mechanically agitated vessels are given by the following empirical expressions:

$$d_{SM} = 4.15 \times 10^{-3}[(\sigma^{0.6}/(P/V_L)^{0.4}\rho_L^{0.2}]H^{1/2} + 0.9 \times 10^{-3}$$

$$H = 4.24[V_s\mu_L H/g\Delta\rho\ d_{SM}^2]^{1/2} + 0.916[(P/V_L)^{0.4}\rho_L^{0.2}/\sigma^{0.6}][V_s\mu_L/g\Delta\rho\ d_{SM}^2]^{1/2}$$

where: d_{SM} = mean bubble diameter, m
σ = surface tension between gas and liquid, mN/m (dyn/cm)
P = mixing power dissipation, kW
V_L = liquid volume, m^3
ρ_L = density of liquid, kg/m^3
V_s = superficial gas velocity, m/s /AL viscosity of liquid, Pa-s
g = acceleration of gravity, M/S^2
$\Delta\rho$ = density difference between gas and liquid phases, kg/m^3

The above two equations must be solved simultaneously and will require the solution of an equation of cubic form. These correlations are based on the gas phase being sparged into the mixing vessel. Gas dispersion from surface entrainment due to votexing, etc., is not included. The mixing power dissipation must be corrected

for gassing in accordance with Figure 26. Two bubble diameter regimes must be considered when calculating the mass transfer coefficient. The first regime applies to bubbles having average diameters greater than 2.5 mm. Such bubbles are typically produced when pure liquids are aerated in mixing vessels or in sieve plate columns. In general, these large bubbles are termed to have unhindered interfacial flow and oscillation. The second regime applies to bubbles of average diameter of less than 2.5 mm. Such small bubbles are typically produced when aqueous solutions or impure liquids are aerated in mixing vessels or when most liquids are aerated with plates having very small perforations. For cases in which the liquid purity is not known, the small bubble regime should be assumed. Correlations for the two regimes are:

$$k_L = 0.42(\mu_L/D_L\rho_L)^{-\frac{1}{2}} (\Delta\rho\mu_L g/\rho_L^2)^{1/3}, \text{ for unhindered interfacial flow}$$

$$k_L = 0.(\mu_L/D_L\rho_L)^{-2/3} (\Delta\rho\mu_L g/\rho_L^2)^{1/3}, \text{ for hindered interfacial flow}$$

where: k_L = liquid phase mass transfer coefficient, m/sec
         D_L = diffusivity of transferring solute in liquid, m²/sec

If the diffusivity, D_L, needed for use in the above equations is not known, it can be estimated from data or methods given in the Perry's Chemical Engineers, Handbook (Section 14 in 4th Edition or Section 3 in 5th Edition). Note that the calculation of the mass transfer coefficients for a given regime involves only physical properties and is independent of agitation conditions.

Calculation of the Performance Equation of a Mixer

The performance equation of a mixer relates mixer size or mixing time to the input and output of the mixing device. The rate of transfer, r_A, incorporates the mass transfer coefficient, k_L, and interfacial area, a, as calculated above. This rate can be used in conjunction with a material balance to relate concentrations of interest to time or size variables.

Three basic fluid contacting patterns describe the majority of gas-liquid mixing operations. These are: (1) mixed gas/mixed liquid - a stirred tank with continuous in and out gas and liquid flow; (2) mixed gas/batch mixed liquid - a stirred tank with continuous in and out gas flow only; (3) concurrent plug flow of gas and liquid - an inline mixer with continuous in and out flow. For these cases the material balance/rate expressions and resulting performance equations can be formalized as:

1) mixed gas/mixed liquid material balance (per unit time):

$$(A \text{ lost by gas}) = (A \text{ gained by liquid}) = (A \text{ transferred})$$

$$F_G (Y_{Ain} - Y_{Aout}) = F_L(X_{Aout} - X_{Ain}) = (-r_A)V$$

where r_A is based on outlet conditions. The performance equation is:

$$V = F_L(X_{Aout} - X_{Ain})/(-r_A) = F_G(Y_{Ain} - Y_{Aout})/(-r_A)$$

2) mixed gas/batch mixed liquid material balance:

(A lost by gas) = (accumulation of A in liquid) = (rate of transfer of A)

$$F_G(Y_{Ain} - Y_{Aout}) = V_L \, dC_A/dt = -r_A \, V$$

The performance of the mixer is based upon integration over the concentration limits of C_{Ao} and C_{Af}:

$$t = V_L/V \int dC_A/-r_A$$

Note that $V_L/V = 1 - H$.

3) plug flow material balance:

$$V = F_L \int dX_A/-r_A = F_G \int dY_A/r_A$$

For dilute systems, the following approximations can be made: $dX_A \approx dC_A/C_U$, and $dY_A \approx dp_A/p_U$. Note the following nomenclature used in the above correlations.

F_G = molar flow rate of inerts in the gas phase, kmol/s
F_L = molar flow rate of inerts in the liquid phase, kmol/s
X_A = moles A/mole inert in liquid phase, dimensionless
Y_A = moles A/mole inert in gas phase, dimensionless
V_L = volume of liquid in vessel, m^3
t = time, s
C_U = concentration of inert components in a phase, mol/m^3
p_A = partial pressure of component A in gas phase, kPa
p_U = partial pressure of inert components in gas phase, kPa

Miscellaneous Mixers

An orifice mixing column is one of the most common and simplest of mixing devices and is often used for caustic washing, water washing, and similar services. It normally consists of two vertical lengths of pipe connected by a return bend at the bottom with orifice plates installed between flanges in the vertical legs. For general caustic wash, water wash, and other easy mixing services, use a mixing energy requirement of about 200 ft-lbs per gallon of total flow and a contact time of about 10 seconds. For difficult services, such as the doctor stages of doctor sweetening plants and the caustic extraction of COS from propane, use a mixing

energy requirement of about 500 ft-lbs per gallon of total flow and a contact time of about 50 seconds. Mixing orifices are normally sized for a pressure drop of 1 to 2 psi each, using for calculation a coefficient of discharge K = 0.65. The size of the orifice selected determines the pipe size and distance between plates which in turn set the amount of contact time available. To arrive at the minimum number of orifices and, consequently, the most economical mixer design, use the maximum pressure drop per orifice (up to 2 psi) compatible with the contact time requirement. The internal diameter of the pipe in which the orifices are installed should be approximately twice the diameter of the orifice. The length of the sections between orifice plates should be approximately three times the internal diameter of the pipe. For water washing in clean services, opposed jet mixing columns are sometimes used instead of orifice mixing columns. Their advantage over orifice mixing columns lies in the fact that only about half as much mixing energy is required for a given mixing efficiency. The mixing column consists of two opposed jet units arranged in series and supported in a vertical column by a flat plate held between a pair of standard flanges. Each unit is composed of two jets having their axes $180°$ apart. A single orifice, taking ½ to 1 psi pressure drop, is provided directly upstream of the first jet unit to premix the phases.

GAS-SOLIDS CONTACTING

Gas-solids contacting is a form of mixing that is done for a variety of reasons, including to effect mass transfer, chemical reactions, drying, and heat transfer applications. Fluidized bed reactors are used in a multitude of applications ranging from drying, to combustion applications, to refinery operations. The various operating ranges of a fluidized bed, as the gas superficial velocity up through it increases, are illustrated in Figure 28. For (A) and (B) the bed is stable, and the pressure drop and Reynolds number (Re) are related. At (B) the pressure drop essentially balances the solids weight. From (B) to (C) the bed of solids is unstable, and particles adjust their position to present as little resistance to flow as possible. At (C) the loosest possible arrangement is obtained in which the particles are in contact. Beyond this point the particles begin to move freely but collide frequently, so that the motion is similar to that of particles in hindered settling. Figure 28 (C) is referred to as the point of fluidization or minimum fluidization. Just beyond this point the particles are all in motion. Increases in Re result from very small increases in ΔP (pressure drop) as the bed continues to expand and the particles move in more rapid, independent motion.

Ultimately, the particles will stream with the fluid and the bed will cease to exist. This occurs in (E). Figure 28 (A) shows a column that is traversed counter gravity-wise by a gas with a superficial velocity, u_d. A pressure drop equal to ΔP_d, will result, and the magnitude of the drop will be determined by the fluid rate and the characteristics of the bed. As the gas velocity is increased, the pressure drop will rise.
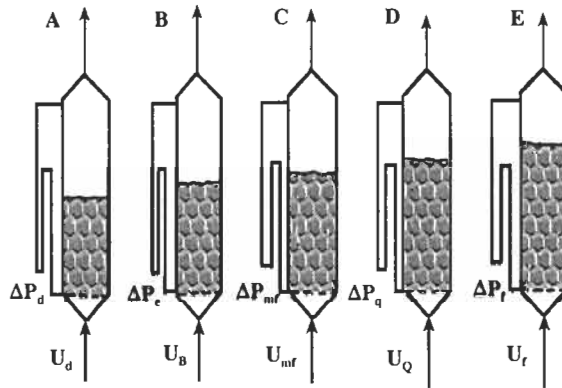
Figure 28. *Operating stages of a dense-phase fluid bed.*
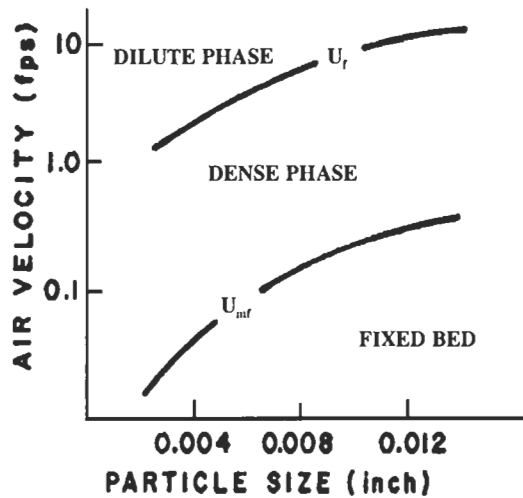
Figure 29. *Velocity ranges encountered in fluidization.*

The order of magnitude over which the gas velocity spans in transforming the bed from incipient fluidization to the dilute phase is shown in Figure 29. Note in Figure 29 that for a particle size (d_p) of 0.002 in., for instance, the velocity range is nearly one hundredfold. As the particle diameter increases, the range narrows considerably. However, even for $d_p = 0.002$ inch it is still about forty-fold. On the average, the ratio of the terminal velocity, U_t, to the minimum fluidization velocity is about 70. Two main types of fluidization have been observed experimentally. In cases in which the fluid and solid densities are not too different with small particles and the gas velocity is low, the bed fluidizes evenly, with each particle moving individually through a relatively uniform mean free path.

The dense phase has many of the characteristics of a liquid. This is called *particulate fluidization*. Where the fluid and solid densities are greatly different or the particles are large, the velocity of flow must be relatively high. In this case fluidization is uneven, and the fluid passes through the bed mainly in the form of large bubbles. These bubbles burst at the surface, spraying solid particles above the bed. Here, the bed has many of the characteristics of a liquid, with the fluid phase acting as a gas bubbling through it. This is called *aggregative fluidization*.

In aggregative fluidization the gas rises through the bed primarily in the form of bubbles. The value of the Froude number ($Fr = U_f^2/d_p g$; in which U_f is the superficial gas velocity, d_p is the particle size, and g is the gravitational constant), is indicative of this type of fluidization. For $Fr > 1.0$, aggregative fluidization is said to prevail. In addition to these classifications, fluidized beds show considerable bypassing and slugging (i.e., intermittent and unstable flow of large gas bubbles through the bed).

Fluidization may be described as incipient buoyancy because the particles are still so close as to have essentially no mobility, whereas the usual desire in fluidization is to create bed homogeneity. Such homogeneity can be achieved only by violent mixing. This is brought about by increasing the fluid velocity to the point of blowing "bubbles" or voids into the bed, which mix the bed as they rise. The increased fluid velocity at which bubbles form first is referred to as the incipient (or minimum) bubbling velocity.

Fine powders can exist over a wide range of bulk densities and, therefore, exhibit substantial differences between incipient buoyancy and incipient bubbling. For coarser granular solids, no distinction can be made between incipient buoyancy and incipient bubbling, as illustrated qualitatively in Figure 30. From a practical point, the incipient bubbling velocity is the more significant one in reactor design. The terms "particulate" and "aggregative" were coined to differentiate between bubbling beds (aggregative) and non-bubbling beds (particulate). In general, liquid-fluidized beds are non-bubbling, whereas gas-fluidized beds bubble. Bubbling is related to fluid and particle properties in a manner permitting the prediction of a system's maximum attainable bubble size, which, if negligible, leads to the observation of so-called particulate fluidization. Rather than employ the terms aggregative and particulate, it is more correct to refer to the maximum stable bubble size for a particular system.

Most gas-fluidized commercial reactors operate under conditions of relatively profuse bubbling, as illustrated in Figure 31. Bubbles form at the ports where the fluidizing gas enters the bed (distributor plate). They form because the velocity at the interface of the bed, just above the entrance hole, represents a gas input rate in excess of what can pass through the interstices at a frictional resistance less than the bed weight. Hence, the layers of solids above the holes are pushed aside until they represent a void through whose porous surface the gas can enter at the incipient fluidization velocity. If the void grows larger, the interface velocity becomes insufficient to hold back the walls of the void.
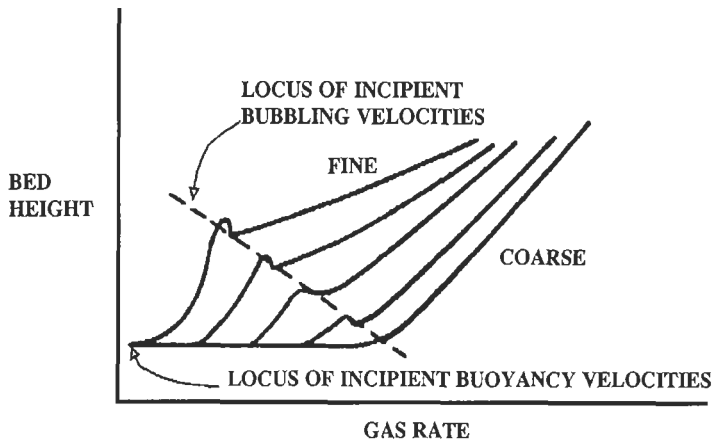
Figure 30. *Particle size effects on minimum fluidization and bubbling velocities.*
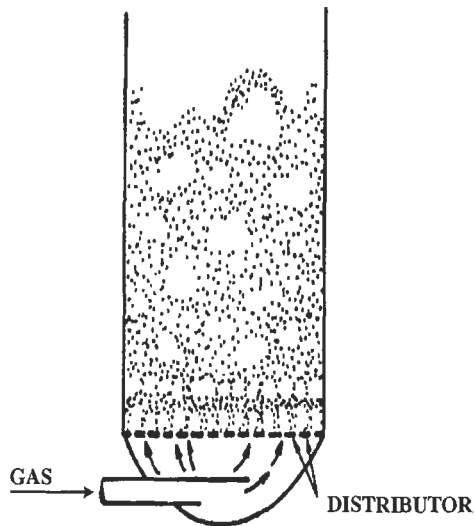
Figure 31. *Freely bubbling gas fluidized bed.*

In the above situation, the walls cave in from the sides, cutting off the void and presenting a new surface to the incoming gas. This sequence is illustrated in Figure 32. The size of the initial bubble resulting from a detached void is typically on the order of about half the penetration depth of the gas jet. Bubbles or gas voids rise in a fluidized bed by being displaced with an inflow of solids from their perimeters.
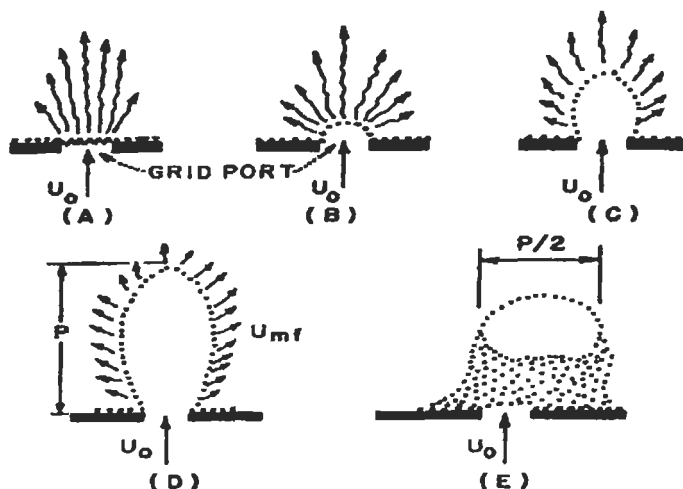
Figure 32. *Sequence of bubble formation.*

The voidage required for the onset of fluidization is termed the minimum gas voidage and is determined readily by subjecting the bed to a rising gas stream and recording the bed height, L_{mf}, that coincides with incipient particle motion. From a knowledge of the buoyant weight of the bed, w, the bed cross section, F, and solids and gas densities, the following relationship is obtained for minimum fluidization voidage:

$$\epsilon_{mf} = 1 - w / [L_{mf} F (\rho_s - \rho_g)]$$

A good indication of fluidization behavior of granular material may be obtained from the pressure drop-flow relationships of the expanded bed. Figure 33 displays ideal fluidization characteristics of a material. Branch ϵ'_e to ϵ_e on the plot pertains to the fixed-bed pressure drop of the bed at a voidage ϵ_e. At ϵ_e expansion occurs and continues along the horizontal line. Particle motion sets in when the voidage has slightly surpassed a value ϵ_{mf} (i.e., the minimum fluidization). Fixed-bed conditions are reestablished as the flowrate is lowered again. This occurs at the pressure drop that is exactly equal to the value that may be calculated from the buoyant weight of the bed, designated as $\Delta\rho_w$.

Very few gas-solid particles follow the behavior presented in Figure 33. The pressure drop-flow relationship in Figure 34 pertains to a system that exhibits channeling. This is an abnormality characterized by the establishment of flow paths in the bed of solids through which large amounts of gas will pass up the column. Branch ϵ'_e - ϵ_e defines the relationship for the fixed bed. The point of initial bed expansion usually will occur at a somewhat higher pressure drop than that calculated from the weight gradient of the bed. As particles "unlock" from each

other, the pressure drop decreases rather suddenly. After the minimum pressure drop at point C has been passed, there is always a pressure drop recovery, but in channeling beds the theoretical value is never quite reached. The amount by which the pressure drop is lower should be indicative of the channeling tendencies of the solids. There are two very common cases of channeling. In through channeling the flow paths extend through the entire bed, and intermediate channeling involves only a portion of the bed. The intermediate channeling bed will yield a higher fluidizing pressure drop with increasing gas rates. Because with channeling the resulting bed density is not homogeneous, there will be local space velocities inside the bed that greatly differ from the overall planned space velocity. This will lead to erratic temperature profiles, as well as inefficiency and larger equipment designs than would be required for a homogeneously operating system. Shape and density of solids are factors that affect channeling, as well as the fluidization chamber diameter.
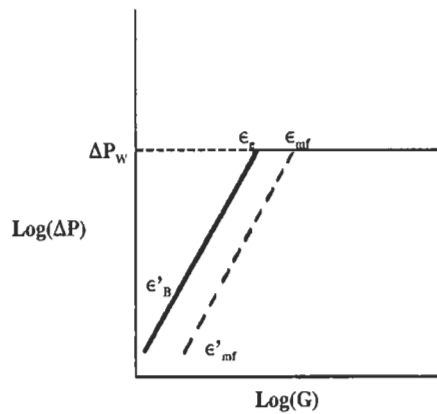
Figure 33. *Pressure drop flow diagram for ideally fluidized bed.*
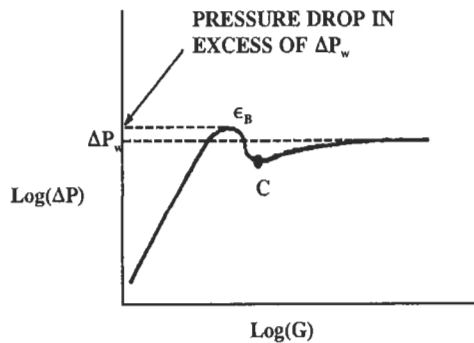
Figure 34. *Pressure drop flow diagram for moderately channeling solids.*

In general, an increase in channeling occurs with decreasing particle size. Furthermore, the design of the gas-inlet device has a profound effect on channeling. With porous plates the gas distribution into a bed tends initially, toward uniformity. Channeling tendencies are always smaller with multi orifice distributors. In this arrangement the gas is introduced through a relatively small number of geometrically spaced holes. Slugging is an abnormality frequently encountered in fluidization. It is considerably affected by the choice and design of the equipment. Characteristics of particles also are involved, although to a lesser extent than in channeling. Slugging can be described as the condition in which bubbles of gas coalesce to a size approaching the order of magnitude of diameter of the confining vessel. Particles form layers, or slugs, between the large gas pockets and tend to move upward in a piston-like manner. On reaching a certain height, they then disintegrate and fall downward in a raining fashion. Local space velocities may differ widely from the overall velocity. This has an erratic effect on yields and temperature distributions. In addition, slugging may accelerate the rate of mechanical attrition of particles. The pressure drop-flow diagram of solid particles reflects the slugging behavior. Referring to Figure 35, the particles approach ideal behavior up to and considerably beyond the point of the onset of fluidization. Beyond a certain flow range, however, the pressure drop will increase above the value calculated from the weight of the bed (point S). This condition is defined as the onset of slugging. The evidence is that the pressure drop excess over the theoretical value is due to friction between the solid slugs and the wall of the fluidized bed. This implies that the height to diameter ratio of the bed is important. The particle size effect on slugging is shown in Figure 36. Smooth operation increases very markedly as particle size decreases. There are numerous correlations in the literature for predicting the onset of slugging, but many of are specific to the type of solids and exact system in which the test is made.
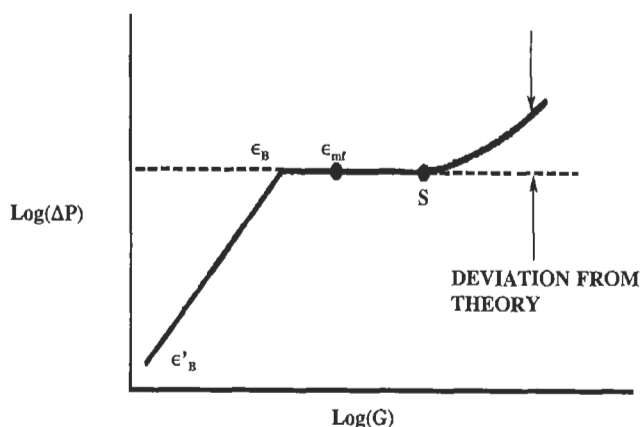
Figure 35. *Pressure drop flow diagram for slugging bed.*
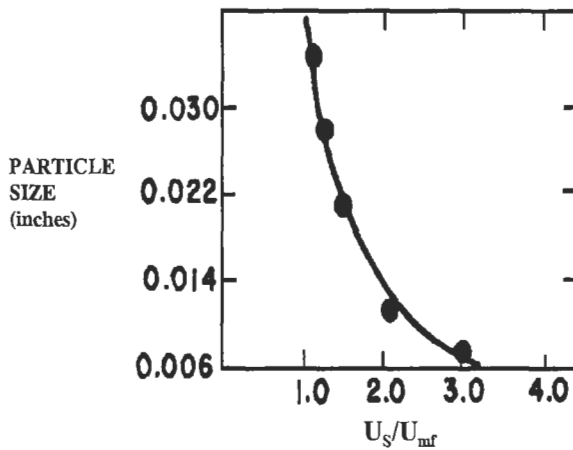
Figure 36. *Relation between particle size and reduced slugging velocities for coal particles.*

The most important application of fluidization is in the field of chemical reaction engineering. Fluidization has evolved to become one of the most useful reactor concepts available in industry. The potential for using fluidized beds as chemical reactors is best illustrated by considering the many applications at the commercial and semi- commercial levels. A convenient classification for gas-fluidized reactors is to consider them as either gas-phase reactors (which may be subdivided into catalytic and noncatalytic), or gas/solid reactors for gas-phase reactions. Examples of catalytic fluidized-bed reactors are petroleum cracking and hydroforming. Examples of gas/solid fluidized-bed reactors are coal combustion, coal carbonization, coal gasification, incineration (oxidation) of waste solids, uranium processing, nuclear materials processing, calcination of ores, etc.

The idealized fluid-bed reactor, a fluidized bed that is not homogeneous, can be considered as having three distinct reaction zones. At the bottom of the bed is the grid region, which contains vertical or horizontal gas jets and/or small formation-sized bubbles; the type of grid will determine the form of the gas voids. Above the grid region is the bubbling zone. In this zone bubbles grow by coalescence and may even approach a diameter similar to the vessel width, forming slugs (typical of laboratory and small pilot-plant reactors). As bubbles break the surface of the bed, particles are entrained in the upward-flowing gas stream. Some of the solids are elutriated, while others fall back to the bed. This is referred to as freeboard region and it affords an opportunity for lean phase reactions. Reactions will occur in the freeboard for most practical situations where moderate to high gas flowrates are employed. The temperature may be higher or lower depending on the heat of the reaction. For example, with the exothermic oxidation reactions occurring in the

freeboard of a coal combustion, gas temperatures 20 °C above the bed temperature are typically observed. The presence of catalyst particles in the freeboard offers additional opportunity for gas conversion.

A special type of fluidized bed is the spouted bed. This technique is used for contacting coarse granular solids. It is generally used for contacting fluids with solids of sizes and other physical properties that render them difficult to fluidize. Spouted beds have been applied to drying materials such as grains and peas, the transportation of solids, humidification control of materials, for heat and mass transfer operations and catalytic reactions. Apparatus construction is of more importance than spouting or fluidization of the solid material. Certain granular solids spout rather than fluidize when they come in contact with a fluid. In this respect, particle size plays a very important role. The motion of solids in a spouted bed is entirely different from that in a freely-bubbling fluidized bed. In the case of a spouted bed, there is an upward movement in the central spout in the form of a dilute fluidized phase and a downward movement in the outside annulus. Spouted beds and channeling fluidized beds are similar from the viewpoint of non-homogeneous fluid distribution over the bed cross section. The spouted bed is a combination of a dilute fluidized phase and a coexistent moving fixed bed of solids. Figure 37 shows a schematic diagram of a spouted bed. The fluid enters the apparatus through an opening in the apex of a conical inlet. The fluid entrance is so abrupt that there is essentially no opportunity for an appreciable lateral distribution over the total apparatus cross section. Hence, a central channel is formed, and in it solids are entrained upward. The solids enter the channel mainly in the cone section.
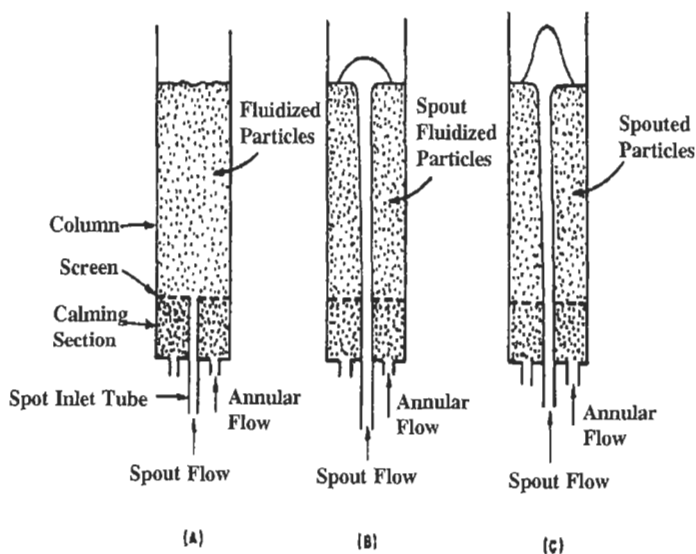
Figure 37. *(A) Fluid bed, annular flow almost entirely at the inlet; (B) Both annular and spout flow at inlet; (C) Spout flow only at inlet.*

The solids concentration in the central channel increases with height and, at the same time, the physical outline of the channel becomes increasingly less distinct. However, for all practical purposes the solids to gas ratio in the central channel is of the same order of magnitude as found in a typical dilute-phase fluidized system. At the upper end of the channel the solids spill over radially into an annulus that defines a column of descending solids. In this ring the solids move downward, without eventually varying their relative positions. The prevailing bed voidage and pattern of solids movement are similar to those found in connection with aerated moving-solids beds. Coexistence of the two phases produces a characteristic solids circulation pattern by which the material is entrained upward in the center and is caused to descend by gravity through the surrounding dense bed contained in the annular space. The fluid distribution over the bed cross section must, of necessity, be highly uniform. This is similar to conditions found in fluidized beds with strong channeling tendencies. In the beds shown in Figure 37, a jet of fluid enters a cylindrical vessel containing the solids along its vertical axis of symmetry. Under the proper conditions the jet penetrates the bed of particles, creating a central spout zone and an annular region surrounding the spout. The gas passes upward through the spout and annulus, while the solids are conveyed up the spout and down the annulus. When the total flow is divided between the spout and annular regions, the bed is called a spout-fluid bed. The spouted and fluidized beds form the asymptotes of the spout-fluid bed.

A centrifugal fluidized bed (CFB) has a number of operating advantages over conventional fluidized beds, which make it attractive for a wide range of applications. A rotating bed is mechanically more complex than a conventional one, however. A CFB is cylindrical in shape and rotates about its axis of symmetry, as shown in Figure 38. As a consequence of the circular motion, the bed material is forced into the annular region at the circumference of the container and "fluid flows radially inward through the porous surface of the cylindrical distributor, fluidizing the bed material against the centrifugal forces generated by the rotation. Radial accelerations many times in excess of gravity can be generated with modest speeds of rotation, permitting much larger gas flowrates at minimum fluidization than are possible with the conventional fluidized bed operating against the vertical force of gravity. The advantages of a CFB over a conventional fluidized bed are as follows:

- The CFB has much higher gas flowrates per unit volume than can be obtained with conventional systems. This results in a smaller distributor surface area and a smaller mass of bed material, reducing the need for a large number of bed modules to achieve large system capacity;
- In applications in which solids need to be fed to the bed continuously, the smaller distributor surface area, cylindrical geometry and rotation of the CFB should lead to fewer solids feed points per unit of capacity than are needed in a conventional bed;
- The CFB is generally more flexible to operate with a faster response time than can be obtained in a conventional fluidized bed. The gas flowrate to the CFB

can be varied over an extremely wide range by adjusting the speed of rotation of the bed and the fluidizing velocity;

- The rotational operation of a CFB leads to a vortex motion in the freeboard which tends to inhibit particle loss by elutriation. Because of the relatively compact nature of the CFB and the operating flexibility provided by the rotational motion, the CFB has been proposed for a variety of applications including coal combustion, flue gas desulfurization, gas combustion, coal liquefaction and food drying.

The term *three-phase fluidization* requires some explanation, as it can be used to describe a variety of rather different operations. The three phases are gas, liquid and particulate solids, although other variations such as two immiscible liquids and particulate solids may exist in special applications. As in the case of a fixed-bed operation, both co-current and counter- current gas-liquid flow are permissible and, for each of these, both bubble flow, in which the liquid is the continuous phase and the gas dispersed, and trickle flow, in which the gas forms a continuous phase and the liquid is more or less dispersed, takes place. A well established device for countercurrent trickle flow, in which low-density solid spheres are fluidized by an upward current of gas and irrigated by a downward flow of liquid, is variously known as the turbulent bed, mobile bed and fluidized packing contactor, or the turbulent contact absorber when it is specifically used for gas absorption and/or dust removal. Still another variation is a three-phase spouted bed contactor.
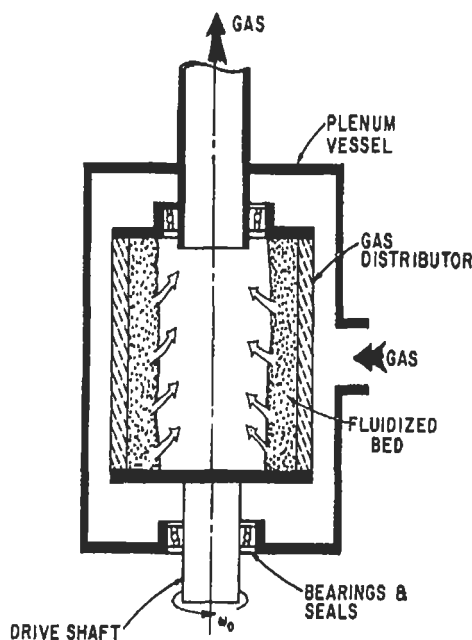
Figure 38. *Centrifugal fluid bed reactor.*

Cocurrent three-phase fluidization is commonly referred to as gas-liquid fluidization. Bubble flow, whether cocurrent or countercurrent, is conveniently subdivided into two modes: mainly liquid-supported solids, in which the liquid exceeds the minimum liquid-fluidization velocity, and bubble-supported solids, in which the liquid is below its minimum fluidization velocity or even stationary and serves mainly to transmit to the solids the momentum and potential energy of the gas bubbles, thus suspending the solids.

Countercurrent bubble flow with liquid-supported solids, which can be affected by downward liquid fluidization of particles having a density lower than that of the liquid, has been referred to as inverse three-phase fluidization. The mass transfer potential of such a countercurrent operation is worthy of study, especially for cases in which dispersion of the gas rather than the liquid is called for and the required gas-liquid ratio and throughput can be effected without flooding. In contrast, the corresponding cocurrent mode has received more attention than all other cases and constitutes the majority of the literature on three-phase fluidization.

The subject of fluidization and research and development in this field, probably reached its peak in the early 1980's with extensive work being applied by the petroleum industry in synthetic fuels development such as shale oil, coal gasification, pyrolysis applications. Prior to this, significant development work was done in the petroleum industry for flexicracking and flexicoking operations. Fluidization still remains an area of active research and industry development, and the technology is well established in a variety of industry sectors.

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RECOMMENDED WEB SITES

1.   *Franklin Miller Inc.*: Crushers, mills, shredders and mixing equipment for chemical processing, waste reduction and wastewater treatment. http://www.franklinmiller.com

2.   *ProQuip, Inc.*: Designers and manufacturers of mixing equipment. http://wwwproquipinc.com

3.   *Lightnin Mixers*: Site describes commercially available mixing equipment and services. http://www.lightnin-mixers.com

4.   *EMI Incorporated*: Manufacturer of fluid mixing equipment, including impeller designs for flow controlled applications in chemical processing, coatings, food, and pharmaceutical. http://www.emimixers.com

Chapter 8
CALCULATIONS FOR
SELECT OPERATIONS

INTRODUCTION

This chapter provides a compendium of short engineering calculation methods and formulas for select process operations and equipment. The formulas and procedures provided in this chapter offer simple, rapid estimates of key parameters important to specifying and obtaining various engineering parameters. The information is derived from the open literature, and is believed to be accurate for obtaining better than an order of magnitude estimate for each calculation.

HEAT CAPACITY RATIOS FOR REAL GASES

For many calculations it is required to know the ratio of C_p/C_v for a gaseous compound. The well-known expression $C_p - C_v = R$ (where R is the universal gas constant) holds good only for gases under ideal conditions. When working with situations involving real gases, the relationship between C_p and C_v is given as:

$$C_p - C_v = R \left(1 + Pr / T_r^2 \left(0.132 + 0.712/T_r\right)^2\right)$$

where: P_r = Reduced pressure = P/P_c
T_r = Reduced temperature = T/T_c
P_c = Critical pressure
T_c = Critical temperature in absolute units
For C_p and C_v in metric units, the value for R is 1.987 cal (gm-mole)-°K

SIZING OF VAPOR-LIQUID SEPARATORS

Vapor-liquid separators (drums) are used to separate a liquid from a vapor-liquid stream with a minimum of liquid carryover. The separator size is determined by the vapor velocity which depends on the entrainment method used. The working equation is:

$$D = \left[(\text{Vapor Load})/0.178 \, R_d\right]^{1/2}$$

where vapor load = cfs of vapor $[\rho_v/(\rho_L-\rho_v)]^{0.25}$. ρ_L and ρ_v are the liquid and vapor densities, respectively, and R_d is a ratio that varies with application. The various values of R_d are summarized below:

Operating Condition	R_d
Usual process application with mesh	1.54
Pressure surges (with mesh)	1.15
Usual process application without mesh	0.2 - 0.44
Knock out drum	0.88

OVERALL EFFICIENCY OF A COMBINATION BOILER

A combination boiler uses more than one fuel, at any given time. A good example is a power boiler in a pulp and paper mill using coal, wood waste, natural gas, and fuel oil. A common calculation method for a boiler burning bark (at 55% moisture), natural gas, and using Bunker C fuel oil is outlined. The method involves one simple step to include a term for coal if needed. Also, the method for computing efficiency is different from the standard methods that involve combustion calculations. The overall method is simpler to use and therefore more appealing to power plant personnel. This simplicity is important to an energy management program because boiler efficiencies should be tracked on a daily basis. We first define the Btu input into the boiler = Btu in fuels + Btu in feedwater; and Btu output from the boiler = Btu in steam out. The overall efficiency is then defined as Btu in steam out/Btu in fuel in and feedwater in. Hence:

$$\text{Eff} = S \times h_s / \{S \times h_w + (\sigma \times 150{,}000) + (g \times 10^3) + (W \times 2000 \times 4000)\}$$

where S = lb/hr steam generated
h_s = enthalpy of steam out, Btu/lb
h_w = enthalpy of feedwater in, Btu/lb
σ = gallons of Bunker C fuel oil/hr
W = tons/hr of bark usage

PUMP HORSEPOWER CALCULATIONS

A rapid method to estimate pump horsepower is by the following formula:

$$HP = GPM \, (\Delta P)/1715(\eta)$$

where   HP = Pump horsepower
        GPM = Gallons per minute of liquid being pumped
        ΔP = Delivered pressure (discharge minus suction), psig
        η = Pump efficiency

Note that the expression can be rearranged to obtain ΔP in terms of other variables. The expression can therefore be used to generate a family of curves of HP versus ΔP with GPM as a parametric variable.

PUMP EFFICIENCY CALCULATIONS

An approximate method to estimate pump efficiencies is based on the following formula. The expression is valid for flows between 100 and 1,000 gpm, and for developed heads between 50 and 300 ft.

$$\eta = 80 - 0.2855F + 3.78 \times 10^{-4}FG - 2.38 \times 10^{-7}FG^2$$
$$+ 5.39 \times 10^{-4}F^2 - 6.39 \times 10^{-7}F^2G + 4 \times 10^{-10}F^2G^2$$

where η = Pump efficiency expressed as percent
        F = Developed head, ft
        G = Flowrate, gpm

LIME KILN PRECOAT FILTER ESTIMATION

A lime kiln is a common piece of equipment used in the chemical process industry for decomposing calcium carbonate into calcium oxide and carbon dioxide. In the pulp and paper industry the slurry, which consists of a mixture of calcium carbonate, water and some inert materials, is filtered to remove a portion of the water. The filter that is employed is a rotary drum filter, commonly called the precoat filter. The fuel consumption in the kiln depends upon the amount of water entering the kiln. A useful calculation that is often made is aimed at determining the fuel savings when the mud solids to the kiln are increased incrementally. The calculation method outlined below is derived on the basis of one minute of operation, and using the mass balance diagram given in Figure 1.

For one minute of operating time, we may state that:

Mass of slurry in = (X gal) (62.4 lm/ft^3) (1ft^3/7.43 gal) = 8.34 X, lb
Mass of solids in = 8.34 SXη/100, lb

In this expression, X is the mud flowrate to the precoat filter, η is the percent solids in the mud to the precoat filter, and S is the specific gravity of the mud.

Mass of water in = 8.34 SX [(100-η)/100]
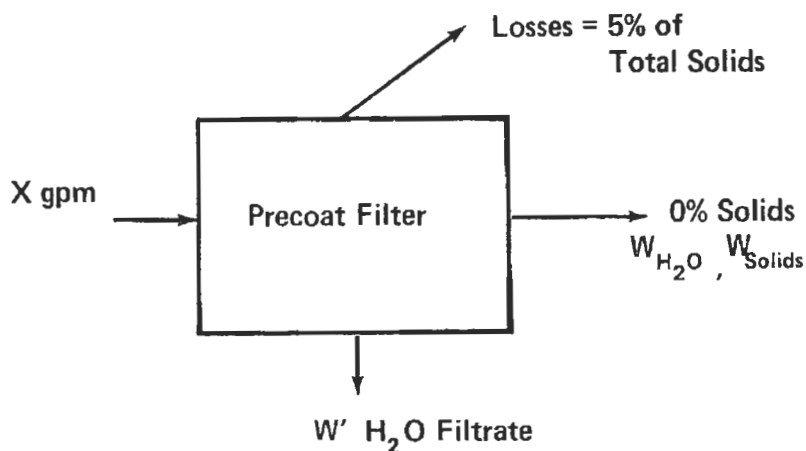
Figure 1. *Material balance diagram for fuel savings in a lime kiln.*

Applying a water balance, we obtain:

$$(8.34)(SX)[1-\eta/100] = W_{H2O} + W'_{H2O}$$

Applying a solids balance, we obtain:

$$(0.95)(8.34SX)\eta/100 = W_{solids}$$

Defining ϕ as the percent solids in the mud from the precoat filter, then:

$$W_{solids}/(W_{H2O} + W_{solids}) = \phi/100$$

Combining the last two expressions, we obtain:

$$\{(0.95)(8.34)SX\eta/100\}/\{W_{H2O} + (0.95)(8.34SX\eta/100)\} = \phi/100$$

and solving for W_{H2O}, we obtain:

$$W_{H2O} = \{(0.95)(8.34)(Sx\eta)/\phi\}\{1-\phi/100\}$$

The Btu needed to evaporate this quantity of water and to raise the vapor to the back-end temperature of the kiln is:

$$\text{Btu/min} = (W_{H2O})(1,1110) + W_{H2O} (t_b - 212)(0.473)$$

where t_b is the temperature in the feed end of the kiln in °F. On a per year basis, the Btu quantity is:

$$\text{Btu/yr} = (\text{Btu/min}) \, (1,440 \text{ min/day}) \, (N \text{ days operation/yr})$$

Hence, the gallons of fuel oil required for the operation are:

$$G = \text{Btu per year}/(150,000 \times 0.85)$$

Combining the above expressions, we obtain:

$$G = 0.08948 \, NSX\eta/\phi[1-\phi/100][1,110 + 0.473(t_b - 212)]$$

The decrease in fuel oil consumption when the solids out decreases from ϕ_1 to ϕ_2 is therefore given by the following expression:

$$\Delta G = 0.08948 \, NSX\eta[(1 - \phi_1/100) \, 1/\phi_1 - (1 - \phi_2/100) \, 1/\phi_2]$$
$$\times \, [1,110 + 0.473(t_b - 212)]$$

This last expression serves as the working formula. Note that natural gas may be substituted for fuel oil, whereby the only resulting change will be the heating value in the derivation.

STEAM SAVINGS IN MULTIPLE EFFECT EVAPORATORS

Multiple-effect evaporators are widely used in the chemical process industry for concentrating dilute solutions of water and a recoverable solute (refer to Chapter 3). A measure of effectiveness of the evaporator set is the steam economy which is expressed in lbs of water evaporated per lb of steam used. Upgrading existing sets or adding new sets affects the steam economy. Any improvement (increase) in the steam economy is beneficial. The question is whether or not the resulting savings will yield an attractive ROI (Return on Investment). The following calculation estimates the change in steam consumption when the steam economy is increased incrementally. The expression derived is applicable to any multiple effect evaporator set. The inputs required are flows, concentrations, density of the dilute solution, and the steam economies. Assuming a basis of one hour of operation, then:

lbs solution into system $= 60 \, G \times d_i$;

lbs solute into system $= 60 \, G \times d_i \, [1-\eta/100]$;

lbs solute out of system $=$ lbs solute into system $= 60 \, G \times d_i \, \eta/100$

lbs of water exiting the system $= 60 \, G \times d_i \, \eta/\phi[1 - \phi/100]$

lbs of water evaporated $=$ lbs of water in - lbs of water out $= 60 \, G \times d_i \, [1 - \eta/100]$ $- (\eta/\phi)[1 - \phi] = 60 \, G \times d_i \, [1 - \eta/100]$;

lbs steam required = 60 G x d_i /S_1 [1 - η/100]

where S_1 is defined as the steam economy prior to upgrading equipment. The pounds of steam required after making changes in equipment is:

lbs of steam = (60 G x d_i)/S_2 [1 - η/100];

Hence, the change in steam requirement is as follows:

ΔS = 60 G x d_i [1 - η/100] [1/S_1 - 1/S_2]

In the above expressions, the parameters are defined as:

η = weight percent solute in the dilute solution

ϕ = weight percent solute in the concentrated solution

S1 = initial steam economy (i.e., lb water evaporated per lb of steam)

S2 = final steam economy (i.e., lb water evaporated per lb of steam).

TEMPERATURE AND LATENT HEAT ESTIMATION FOR SATURATED STEAM

The following expressions can be used to estimate the temperature and enthalpy of steam. The expressions are based upon multiple regression analysis. The equation for temperature is accurate to within $\pm$ 1.5 % at 1,000 psia. The expression for latent heat is accurate to within $\pm$ 3 % at 1,000 psia. Input data required to use these equations is the steam pressure in psia. The parameters in the equations are defined as t for temperature in °F, H_v for latent heat in Btu/lb, and P for pressure in psia.

$$t = 1.8 \exp\{4.61 + 0.250336 (\ln(P) - 2.686)\} + 32$$

$$H_v = 1.8 \{\exp(6.29517 - 0.0343248 (\ln(P) - 2.686)) - 0.116672P\}$$

ESTIMATING CONDENSATE FOR FLASH TANKS

Often in plant operations condensate at high pressures are let down to lower pressures. In such situations some low-pressure "flash" steam is produced, and the low-pressure condensate is either sent to a power plant or is cascaded to a lower pressure level. The following analysis solves the mass and heat balances that describe such a system, and can be used as an approximate calculation procedure. Refer to Figure 2 for a simplified view of the system and the basis for developing the mass and energy balances. We consider the condensate to be at pressure P_1 and temperature t_1, from whence it is let down to pressure P_2. The saturation temperature at pressure P_2 is t_2. The vapor flow is defined as V lbs/hr, and the condensate quality is defined as L lbs/hr. The mass balance derived from Figure 2 is:

$$F = V + L$$
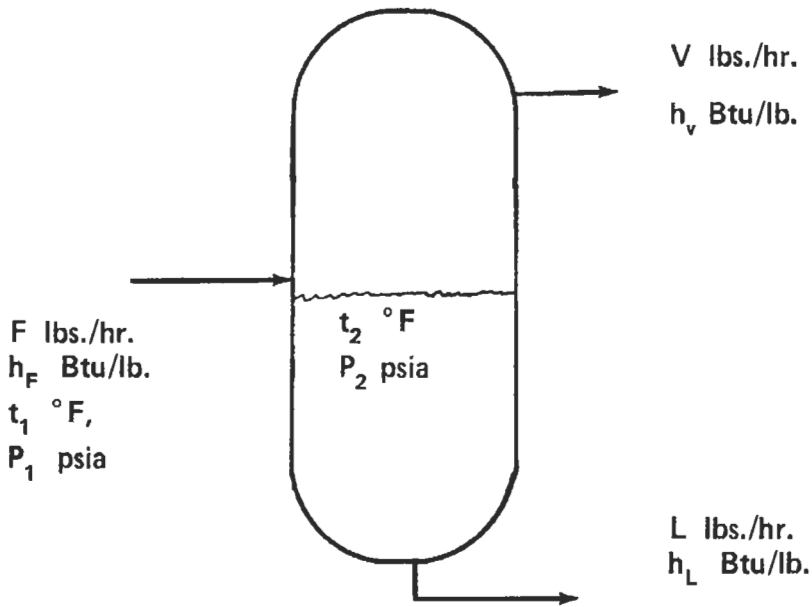
Figure 2. *Material and energy balance diagram for flash tank.*

And the heat balance is:

$$F \times h_F = V \times h_v + L \times h_L$$

where we define $h_F = t_1 - 32$, and $h_L = t_2 - 32$. The expression may be restated as:

$$F(t_1 - 32) = Vh_v + L(t_2 - 32)$$

Combining the above expressions, we obtain:

$$L/F = \{h_v - t_1 + 32\}/\{h_v - t_2 + 32\}$$

This equation expresses the fraction of the high-pressure condensate returned as low-pressure condensate. The input information needed to apply the working equation are t_2 and P_1. Values for h_v can be approximated from the following:

$$h_v \approx \Delta h_v + h_L = \Delta h_v + t_2 - 32$$

Combining this approximation into the working expression gives:

$$L/F = (\Delta h_v + t_2 - t_1)/\Delta h_v$$

To summarize, the symbols used in the development of the expression are defined as follows:

F = Flow rate of high-pressure condensate, lbs/hr

h_v = Enthalpy of the vapor, Btu/lb

h_F = Enthalpy of high-pressure condensate, Btu/lb

h_L = Enthalpy of low-pressure condensate, Btu/lb

L = Flowrate of low pressure condensate, lb/hr

$P1$ = Upstream pressure, psia

$P2$ = Pressure downstream of valve, psia

t_1 = Temperature upstream of letdown valve, °F

t_2 = Temperature downstream of letdown valve, °F

Δh_v = latent heat of vaporization at temperature t_2, Btu/lb.

LINEAR VELOCITY OF AIR THROUGH DUCTS

An estimate of the linear velocity of air through ductwork is a common exercise for process engineers. The calculation requires information on the static pressure drop and volumetric flowrate at any given temperature and pressure.

A simplified estimate can be made by first converting the flow at actual conditions to the flow at standard conditions (i.e., at 70 °F and 1 atm). The calculation basis for the linear velocity assumes a roughness coefficient of 0.0005 and a kinematic viscosity for air of 1.62×10^{-4} ft²/sec. From the ideal gas law, the following expression is developed:

$$\text{scfm} = (\text{acfm}) (P/T) (529)$$

where: scfm = flow, ft³/min at 70 °F and 1 atm

acfm = flow, ft³/min at T °R and 1 atm

P = actual pressure, atm

T = absolute temperature, °R

From the standard cubic feet per minute estimate, the linear velocity is as follows:

$$V = 466 \ (h^{0.4}) \ (\text{scfm})^{0.24}$$

Where V is the linear velocity in fpm and h is the static pressure drop in units of inches of water per 100 ft of pipe.

THERMAL CONDUCTIVITIES OF GASES

Thermal conductivities for gaseous compounds are important in unit operations involving heat transfer coefficients. Thermal conductivities can be readily computed from an empirical polynomial expression that has the following form:

$$K_G = A + BT + CT^2 + DT^3$$

where K_G = Thermal conductivity of the gas at low pressure, (approximately 1 atm). A, B, C, and D are correlation constants that are specific to the chemical, and T is temperature, °K. Correlation constants for different chemical compounds are given in Table 1.

Table 1 provides states the temperature range over which the correlation constants are reported from the literature. Estimates from this expression using the constants in Table 1 are generally accurate and typically provide agreement to well within ± 1% compared to experimentally measured thermal conductivities.

More extensive data can be found in standard references such as Perry's *Chemical Engineering Handbook* or the *CRC Handbook of Chemistry and Physics*. The following table, however will provide some basic information for many common calculations that the process engineer can easily run on his or her PC.

Table 1. *Correlation Constants for Estimating Thermal Conductivity of Gases*

Chemical	A	B x 10^2	C x 10^4	D x 10^8	Range, °C
Halogens					
Flourine, F_2	1.8654	19.79	1.24	-17.77	-30 to 525
Chlorine, Cl_2	3.25	5.8	0.21	-1.25	-80 to 1,200
Bromine, Br_2	-0.16	4.13	-0.03	-0.09	-80 to 1,200
Iodine, I_2	0.63	2.73	-0.03	0.15	-80 to 1,200
Sulfur oxides					
Sulfur dioxide, SO_2	-19.31	15.15	-0.33	0.55	0 to 1,400
Sulfur trioxide, SO_3	-15.96	16.9	-0.47	3.00	-100 to 1,000
Nitrogen oxides					
Nitrous oxide, N_2O	-18.71	21.26	-0.2142	-0.6371	-100 to 1,400
Nitric oxide, NO	11.99	17.18	-0.02	-0.85	-190 to 1,400
Nitrogen dioxide, NO_2	-33.52	26.46	-0.755	1.071	25 to 1,400
Carbon oxides					
Carbon monoxide, CO	1.21	21.79	-0.8416	1.958	-160 to 1,400

Table 1 Continued.

Chemical	A	B x 10^2	C x 10^4	D x 10^8	Range, °C
Carbon dioxide, CO_2	-17.23	19.14	0.1308	-2.514	-90 to 1,400
Hydrogen halides					
Hydrogen fluoride, HF	9.21	12.60	0.54	-2.34	-100 to 1,400
Hydrogen chloride, HCl	-0.26	12.67	-0.25	0.16	-150 to 1,400
Hydrogen bromide, HBr	-1.89	9.16	-0.26	0.53	-150 to 1,400
Hydrogen iodide, HI	-5.14	7.28	-0.22	0.43	-150 to 1,400
Nitrogen hydrides					
Ammonia, NH_3	0.91	12.87	2.93	-8.68	0 to 1,400
Hydrazine, N_2H_4	-53.89	28.50	0.20	-0.19	0 to 1,400
Hydrogen oxides					
Water, H_2O	17.53	-2.42	4.3	-21.73	0 to 800
Hydrogen peroxide, H_2O_2	-21.07	16.97	0.17	-1.56	0 to 1,200
Diatomic gases					
Hydrogen, H_2	19.34	159.74	-9.93	37.29	-160 to 1,200
Nitrogen, N_2	0.9359	23.44	-1.21	3.591	-160 to 1,200
Oxygen, O_2	-0.7816	23.8	-0.8939	2.324	-160 to 1,200
Inert gases					
Helium, He	88.89	93.04	-1.79	3.09	-160 to 800
Neon, Ne	21.75	36.81	-2.005	6.042	-160 to 1,200
Argon, Ar	6.48	13.23	-0.52	1.32	-160 to 1,200
Olefins					
Ethylene, C_2H_4	-42.04	28.65	0.7963	-3.262	-75 to 1,000
Propylene, C_3H_6	-18.11	14.57	2.38	-9.17	-100 to 1,000
1-Butene, C_4H_8	-25.12	13.78	2.43	-10.20	-100 to 1,000

Table 1 Continued.

Chemical	$\underline{A}$	$\underline{B \times 10^2}$	$\underline{C \times 10^4}$	$\underline{D \times 10^8}$	$\underline{\text{Range, }^\circ C}$
		Alkanes			
Methane, CH_4	-4.463	20.84	2.815	-8.631	0 to 1,000
Ethane, C_2H_6	-75.8	52.57	-4.593	39.74	0 to 750
Propane, C_3H_8	4.438	-1.122	5.198	-20.08	0 to 1,000
		Xylenes			
o-Xylene, $C_6H_4(CH_3)_2$	-13.66	8.53	1.78	-6.26	0 to 1,000
m-Xylene, $C_6H_4(CH_3)_2$	31.53	-10.02	3.97	-14.58	0 to 1,000
p-Xylene, $C_6H_4(CH_3)_2$	-19.53	9.29	1.81	-6.93	0 to 1,000
		Aromatics			
Benzene, C_6H_6	-20.19	8.64	2.34	-9.69	0 to 1,000
Naphthalene, $C_{10}H_8$	-22.40	11.79	0.91	-2.54	0 to 1,000
		Benzene derivatives			
Aniline, $C_6H_5NH_2$	-26.39	11.89	1.55	-4.30	0 to 1,000
Phenol, C_6H_5OH	-31.87	15.26	1.74	-4.40	0 to 1,000
Chlorobenzene, C_6H_5Cl	-15.27	6.29	1.75	-5.53	0 to 1,000
		Alkyl aromatics			
Toluene, $C^6H_5CH_3$	18.14	-9.57	5.66	-22.22	0 to 1,000
Ethylbenzene, $C_6H_5C_2H_5$	1.44	-1.40	5.11	-21.31	0 to 1,000
Cumene, $C_6H_5CH(CH_3)_2$	-13.35	5.38	4.33	-17.92	0 to 1,000
		Cycloalkanes			
Cyclopropane, C_3H_6	-20.46	9.74	3.77	-16.28	0 to 1,000
Cyclobutane, C_4H_8	-23.39	9.15	3.52	-14.81	0 to 800
Cyclohexane, C_6H_{12}	-20.57	4.45	4.07	-17.31	0 to 800
Cyclopentane, C_5H_{10}	-20.35	5.91	3.87	-16.51	0 to 800

DETERMINING PSEUDOCRITICAL PROPERTIES

Critical properties of gaseous compounds are useful in determining the P-V-T (*Pressure-Volume-Temperature*) properties at nonideal conditions. The compressibility factor Z is defined by the following relationship:

$$PV = nZRT \quad PV = nZRT$$

where n is the number of moles and R is the universal gas law constant. To calculate Z, the critical properties must be known. Knowing the critical pressure and temperatures, charts for computing Z can be used.

When the compounds involved are gaseous mixtures, a weighting method is used to arrive at the critical properties of the mixture.

Table 2 illustrates the weighting method that can be used. The required input of parameters to perform computations are the number of components, the mole fraction of ith component, the critical pressure of ith component in units of atm, the critical temperature of the ith component (°K), the molecular weight of the ith component, the pressure of the mixture (atm), the temperature of the mixture (°F), and the compressibility factor of the mixture, Z_m.

The example provided in Table 2 is for a four component mixture of hydrocarbons (methane, ethane, propane, and n-octane). The weighting method is a common calculation procedure that process engineers will encounter many times. Computations for simple systems can be easily set up on an Excel spreadsheet.

Table 2. *Weighting Method for Estimating Critical Properties of Organics*

Compound	Mole Fraction	Critical Press. (atm)	Critical Temp. (°R)	Pseudocritical Press. [a]	Pseudocritical Temp. [b]
Methane	0.20	45.4	190.6	9.080	38.12
Ethane	0.30	48.2	305.4	14.460	91.62
Propane	0.35	41.9	369.8	14.670	129.43
n-Octane	0.15	24.5	568.8	3.675	85.32
				41.885	344.49
P_c (mixture) = 41.885 atm.					
T_c (mixture) = 344.490 °K					

(a) ...Col. 2 x Col. 3; (b) ...Col. 2 x Col. 4.

The following formulas can be used to calculate critical properties:

$$T_{cm} = \sum T_{ci} y_i$$
$$P_{cm} = \sum Pc_i y_i$$
$$M_m = \sum M_i y_i$$
$$T_{rm} = T_m / T_{cm}$$
$$P_{rm} = P_m / P_{cm}$$
$$V_r = Z_m R T_{cm} / P_{cm} M_m$$

In the above formulas the terms are defined as follows:

T_m = Temperature of the mixture, °R

T_{cm} = Critical temperature of the mixture, °R

T_{ci} = Critical temperature of the ith component, °R

P_m = Pressure of the mixture, atm

P_{cm} = Critical pressure of the mixture, atm

P_{ci} = Critical pressure of mixture, atm

M_m = Molecular weight of mixture

M_i = Molecular weight of ith component

y_i = Mole fraction of ith component

R = Universal gas law constant = 0.73 (atm)(ft^3)/(lb-mol)(°R)

T_{rm} = Reduced temperature of mixture

P_{rm} = Reduced pressure of mixture

Z_m = Compressibility factor of mixture

ESTIMATING HEAT EXCHANGER TEMPERATURES

Chapter 1 provides a summary of important equations for estimating the terminal temperatures in a heat exchanger. Here we formalize a short estimating procedure for a countercurrent flow situation. Assume that a specifier of a heat exchanger has defined a preliminary sizing of the unit. The system requires heat and material balances.

The only information available are the inlet temperatures of the hot and cold fluids and estimates for the overall heat transfer coefficient U and the heat transfer surface A. The flows are fixed and the specific heats of the fluids known.

The problem is different from typical heat transfer problems. The heat balance is not straightforward because the outlet temperatures are unknown. A trial-and-error procedure is therefore required.

Before this procedure is set up, the convergence equations have to be established as follows (Refer to Figure 3):

$$W_1 C_1 (T_1 - t_1) = W_2 C_2 (T_2 - t_2) = UA \, \Delta T_{LMTD}$$

where W_1 and W_2 are mass flows of the cold and hot fluid in lbs/hr, respectively.

Parameters C_1 and C_2 are specific heats of the cold and hot fluids in Btu respectively, and the log-mean temperature difference is defined as follows:

$$\Delta T_{LMTD} = \{(T_2 - T_1) - (t_2 - t_1)\}/\{\ln[(T_2 - T_1)/(t_2 - t_1)]\}$$

Rearranging this expression gives the following:

$$(W_1C_1/W_2C_2)(T_1 - t_1) = T_2 - t_2$$

$$T_2 = T_2 - aT_1 + at_1 = (T_2 + at_1) - aT_1$$

where $a = W_1C_1/W_2C_2$. Defining the following parameter and substituting into the above expression, $b = T_2 + at_1$:

$$\Delta T_{LMTD} = \{(T_2 - T_1) - (b - aT_1 - t_1)\}/\ln[(T_2 - T_1)/(b - aT_1 - t_1)]$$

The heat transfer rate can be determined from the following equation:

$$Q = UA\ [(T_2 - T_1) - (b - aT_1 - t_1)] / [\ln\{(T_2 - T_1) / (b - aT_1 - t_1)\}]$$

The above working expression may also be stated in the following form:

$$W_1C_1\ (T_1 - t_1) = UA\ [(T_2 - T_1) - (b - aT_1 - t_1)] / [\ln\{(T_2 - T_1) / (b - aT_1 - t_1)\}]$$

And upon simplifying further, we obtain:

$$W_1C_1\ (T_1 - t_1)\ [\ln(T_2 - T_1) / (b - aT_1 - t_1] = UA\ (T_2 - T_1) - (b - aT_1 - t_1)$$
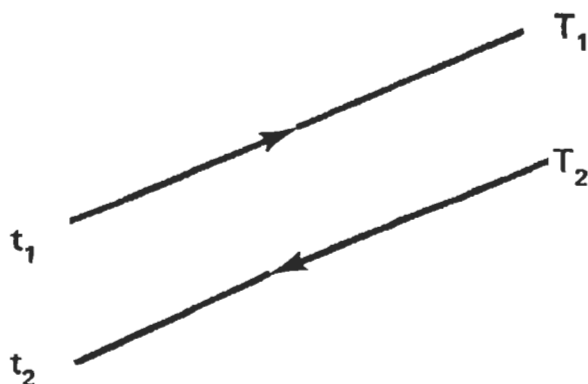
Figure 3. *Terminal temperatures in a counterflow heat exchanger.*

The last expression represents the working formula, whereby the calculation method assumes a value of T_1, calculating both sides of the above expression until they converge.

ESTIMATING THE VISCOSITY OF GASES

The following polynomial can be used to estimate the viscosity of gases:

$$\mu = A + BT + CT^2$$

where: μ = Gas viscosity, micropoise, and A, B, and C are constants. The calculation procedure used here is similar to that explained in the section on estimating thermal conductivity. The only change is the degree of the polynomial, which is now two. Table 3 provides reference viscosity data for gases at 25 °C, and Table 4 provides correlation constants for various common gases encountered throughout the CPI. The viscosities of mixtures can be computed from the following relation:

$$\mu_m = \sum y_i \mu_i \, (M_i)^{1/2} \, / \, \sum y_i \, (M_i)^{1/2}$$

where: μ_m = Viscosity of mixture
μ_i = Viscosity of the ith component
y_i = Mole fraction of the ith component
M_i = Molecular weight of the ith component

The summations are done from the ith to N number of components. The reader will encounter many situations where it is necessary to estimate the viscosities of mixtures. This is especially the case when calculations involve heat transfer coefficients which requires knowledge of the viscosity of fluids involved. Unlike estimates for densities, viscosity functions tend to be non-linear. The formula provided for estimating the viscosity of a mixture is only reliable for low pressures, and should not be applied to pressures above 100 atm. The term viscosity is a fundamental physical property that has great importance in fluid dynamics as well as heat transfer. Its definition stems from the property of any fluid. A fluid is a substance which undergoes continuous deformation when subjected to a shear stress. The resistance offered by a real fluid to such deformation is called its consistency. If τ (lb. force/ft^2) is the shear stress which maintains at a point in a fluid the time rate of shearing strain du/dy (i.e., velocity gradient, (ft/sec)/ft perpendicular to the direction of τ), the numerical measure of the consistency is the quantity τg_c/ du/dy [lb-mass / (sec)(ft)] in which g_c is the dimensional constant 32.1740 (lb-mass)(ft)/(lb-force)(sec^2). For gases and for Newtonian liquids the consistency is constant if the static pressure and temperature are constant or fixed;

for such substances the consistency is usually called the viscosity (= absolute viscosity). If two parallel plates are separated by a thin layer, L ft. thick, of a fluid of viscosity μ lb-mass/(sec)(ft), the force τ per unit area (lb-force/ft^2) which must be exerted in order to keep one plate moving, parallel to the other at a relative velocity of 1 ft/sec is $\tau = \mu/g_c L$. If the consistency of a fluid is not constant but is, at constant temperature, a function only of the shear stress τ, the fluid is called a complex (or non-Newtonian) fluid. An ideal or perfect fluid is a hypothetical liquid or gas which offers no resistance to shear and therefore has zero consistency. Such an imaginary perfect fluid is not to be confused with a "perfect gas," which is a fluid for which $PV = RT$, where P is the absolute static pressure, V is the specific volume, R is the gas constant, and T is the absolute temperature. In many cases highly incorrect results are obtained if the viscosity is neglected, although in the same problem the relation PV-RT may be safely used. The unit of viscosity (i.e., absolute viscosity) is the poise (poise = 1 (dyne)(sec)/cm^2 = 1 g/(sec)(cm)). Viscosity data are normally tabulated in centipoises (cp) or micropoises as in Table 3. In the absolute system of units, the dimensionless constant g_c is simply unity.

Table 3. *Viscosity (micropoise) of Common Gases at 25 °C*

Compound	μ_G	Compound	μ_G	Compound	μ_G
Flourine	232.6	Hydrogen iodide	178.2	1-Butene	74.3
Chlorine	133.5	Ammonia	103	Methane	111.9
Bromine	153.8	Hydrazine	80.13	Ethane	92.2
Iodine	136.5	Water	90.14	Propane	82.4
Sulfur dioxide	128.2	Hydrogen peroxide	95.2	o-Xylene	64.8
Sulfur trioxide	138.6	Hydrogen	84.7	m-Xylene	56.7
Nitrous oxide	158.2	Nitrogen	169.5	p-Xylene	58.4
Nitric oxide	192.8	Oxygen	199.2	Benzene	74.6
Carbon monoxide	165.2	Helium	195.7	Naphthalene	53.2
Carbon dioxide	153.4	Neon	306.5	Toluene	68.8
Hydrogen flouride	110.8	Argon	223.3	Ethylbenzene	63.2
Hydrogen chloride	114.2	Ethylene	101.2	Cumene	58.8
Hydrogen bromide	184.5	Propylene	83.9	Phenol	79.0

Table 4. *Correlation Constants for Estimating Gas Viscosity*

Compound	A	B x 10^2	C x 10^6	Appl.Temp. Range,°C
Flourine	22.09	76.9	-211.6	-200 to 1,000
Chlorine	5.175	45.69	-88.54	-200 to 1,200
Bromine	2.153	54.50	-122.2	-200 to 1,200
Iodine	-17.75	54.71	-99.70	-200 to 1,200
Sulfur dioxide	-3.793	46.45	-72.76	-100 to 1,400
Sulfur trioxide	4.207	47.12	-68.34	-100 to 1,400
Nitrous oxide	32.28	44.54	-77.08	-150 to 1,400
Nitric oxide	56.77	48.14	-84.34	-150 to 1,400
Carbon monoxide	32.28	47.47	-96.48	-200 to 1,400
Carbon dioxide	24.45	45.49	-86.49	-100 to 1,400
Hydrogen fluoride	-19.21	45.98	-79.96	-120 to 1,400
Hydrogen chloride	-9.554	54.45	-96.56	-120 to 1,400
Hydrogen bromide	-23.37	74.03	-144.8	-120 to 1,200
Hydrogen iodide	-17.65	69.77	-136.5	-120 to 1,400
Ammonia	-9.372	38.99	-44.05	-200 to 1,200
Hydrazine	-17.05	34.01	-47.51	-200 to 1,400
Water	-31.89	41.45	8,272	0 to 1,000
Hydrogen peroxide	5.381	28.98	38.40	-80 to 1,000
Hydrogen	21.87	22.2	-37.51	-160 to 1,200
Nitrogen	30.43	49.89	-109.3	-160 to 1,200
Oxygen	18.11	66.32	-187.9	-160 to 1,000
Helium	54.16	50.14	-89.47	-160 to 1,200
Neon	87.79	78.60	-176.2	-160 to 1,200
Argon	43.48	63.99	-128	0 to 1,200
Ethylene	3.586	35.13	-80.55	-100 to 800

Note that the kinematic viscosity of a fluid is defined as the ratio of its viscosity to the fluid density. The c.g.s. unit of kinematic viscosity is usually called the stoke, and is equal to 1 cm^2/sec.

ESTIMATE FOR MECHANICAL DESUPERHEATERS

The chemical process industry relies on mechanical desuperheaters when there is a need to reduce the temperature of superheated steam. One way of accomplishing this is by injecting feedwater quality water or condensate into the steam header at about 100 psi greater than the header pressure. The design of the superheater is based on material and energy balances. Important parameters to be specified are the steam flows into the unit, and the temperatures of the steam in and out of the system. The material and energy balance diagram for a mechanical desuperheater is shown in Figure 4. The following parameters are defined:

h_1 = Enthalpy of steam in
$h_1 m_1$ = Total Btu in
$h_2 m_2$ = Total Btu out in steam
$h_1 m_1 - h_2 m_2$ = Difference in Btu

The difference is the heat that is picked up by the injected water, and hence we may write:

$$m_w[C_p(t_1 - t_2) + L_H] = m_1 h_1 - m_2 h_2$$

From a mass balance we write:

$$m_2 = m_1 + m_w$$

Combining the above expressions and solving for the mass of desuperheating water used, we arrive at the following working expression:

$$m_w = m_1(h_1 - h_2) / [C_p(t_1 - t_2) + L_H + h_2]$$
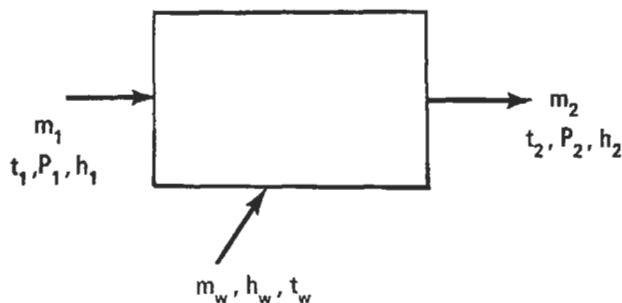
Figure 4. *Mass and energy balance for desuperheater system.*

In the above analysis the parameters are as follows:

C_p = Specific heat of the desuperheating water, Btu/lb-°F

h_1 = Enthalpy of superheated system, Btu/lb

h_2 = Enthalpy of saturated steam, Btu/lb

L_H = latent heat of vaporization of water at t_w, Btu/lb

m_1 = Mass of superheated steam into the system, lb/hr

m_2 = Mass of saturated steam out, lb/hr

m_w = Mass of desuperheating water used, lb/hr

t_1 = Temperature of superheated steam, °F

t_2 = Temperature of saturated steam, °F

t_w = Temperature of desuperheating water, °F

ESTIMATING PUMP HEAD WITH NEGATIVE SUCTION PRESSURE

Figure 5 illustrates the usual configuration for determining the total head developed by a pump working with suction pressure below atmospheric conditions. Suction head is measured by a mercury U-tube which is connected to the suction through an air-filled tube. Discharge head is measured by means of a Bourdon gage connected to the pump discharge through a water-filled tube. The working equations are for the head and the horsepower requirements:

$$H = 144P_g /\gamma + \gamma_m h_{sg} + Z_d + Z_s + 0.00001056 \,(gpm) \,(d_d^2 - d_s^2)$$

$$HP = (gpm) \times (H) \times (\zeta) / 3960$$

where: gpm = Fluid flow rate

ζ = Specific gravity of the liquid

H = Total pump head, ft

P_g = Discharge gage reading, psi

γ = Specific weight of liquid being pumped, lb/ft^3

  = (32.2) (specific gravity of liquid)

γ_m = Specific weight of mercury, lb/ft^3

  = (32.2) (specific gravity of mercury)

Z_d, Z_s = Elevation at discharge and suction, respectively

gpm = Flow rate of liquid being pumped, gal/min

d_d = Diameter of discharge pipe, in.

d_s = Diameter of suction pipe, in.

The reader should refer to the chapter on pumps for more detailed calculation methods and for a description of common pumping equipment and pumping applications. The simple relationships provided here are useful in obtaining initial design specifications information in sizing a pump for an application.
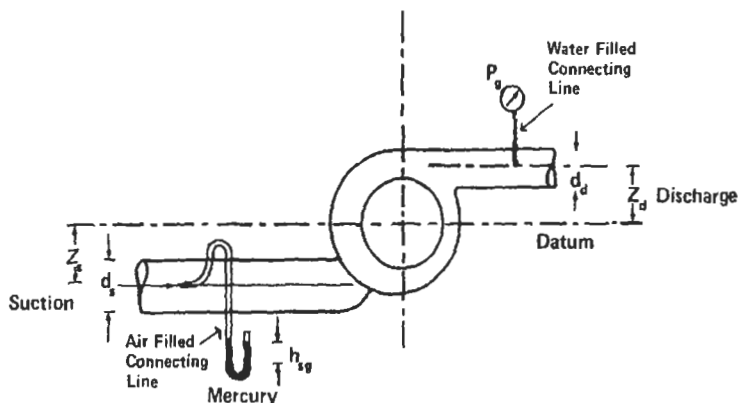
Figure 5. *Diagram for determining head for a pump with negative suction pressure.*

CALCULATIONS FOR BACK-PRESSURE TURBINES

Back-pressure power is achieved by letting down steam at high pressure and temperature for process use. Since process demands change with time and plant conditions, it is of interest to investigate how much power can be obtained at the generator terminals for given quantities of extraction and exhaust steam. Figure 6 illustrates a generalized steam flow configuration for a double-extraction condensing turbine. The outlet enthalpies are measured upstream of any desuperheating stations. The condensing cooling water removes an amount of heat that is equal to $(W_3 \times h_3)$ Btu/hr. The working equations for this analysis are:

$$W = W_1 + W_2 + W_3$$

$(3143)/(h_i - h_1) =$ (Theoretical lbs of steam)/(kW generated at 1st extraction point)

$(3143)/(h_i - h_2) =$ (Theoretical lbs of steam)/(kW generated at 2st extraction point)

$(3143)/(h_i - h_3) =$ (Theoretical lbs of steam)/(kW generated at condensing section)

Since each section will have a certain isoentropic efficiency, the actual steam rate for each will be different. The actual steam rates can be expressed by the formulas presented below.
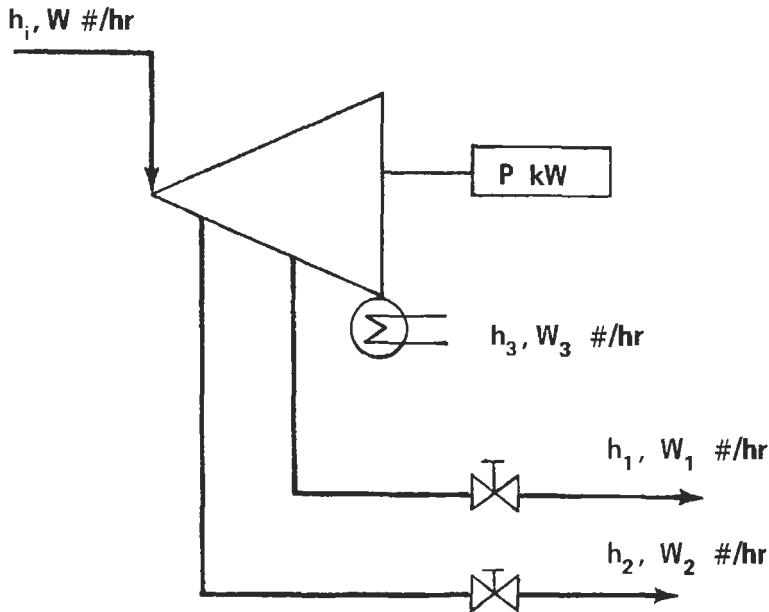
Figure 6. Simplified diagram for back-pressure turbine analysis.

First extraction point = (3143) / [(h$_i$ - h$_1$)e$_1$]

Second extraction point = (3143) / [(h$_i$ - h$_2$)e$_2$]

Condensing stage = (3143) / [(h$_i$ - h$_3$)e$_3$]

From the above, the total output less generator loss is expressed by the following:

(h$_i$ - h$_1$)e$_1$W$_1$/(3143) + e$_2$W$_2$(h$_1$ - h$_2$)/(3143) + e$_3$W$_3$(h$_1$ - h$_3$)/(3143) = P/e$_g$

In the above working equation parameter e$_g$ denotes the generator efficiency which usually reaches around 98 %. Note that a first pass estimate can be made by assuming e$_1$, e$_2$, and e$_3$ to remain constant. A more rigorous analysis can be made by varying these efficiencies using data supplied by the equipment supplier. The initial information required for the analysis are the flows, the efficiencies, and the enthalpies. The outputs from the calculations are the total kW at the generator terminals and the overall water rate for the machine This type of analysis is particularly well suited for sensitivity studies of the type used in determining steam-power balances in an operating facility.

TUBESIDE FOULING RATES IN HEAT EXCHANGERS

A well-known empirical formula for fouling in tubes is:

$$R_t = R_o(1 - e^{-At})$$

where: R_t = Fouling resistance at time t, hr-ft^2-°F/Btu
R_o = Asymptotic value of fouling resistance, hr-ft^2-°F/Btu
t = Time corresponding to the fouling resistance R_t
A = Constant describing the rate of fouling, (time)$^{-1}$

A heat exchange surface becomes fouled when the material deposited on it resists the passage of heat and reduces the conductive part of the heat transfer. When this occurs in practice, the exchanger is taken off line and cleaned until it fulfills the heat duty for which it was designed. It is useful to know the frequency at which the cleaning must be performed. If the downtime for the exchanger is minimized, the operating costs for the system are minimized. The following method uses a trial-and-error procedure that predicts fouling rates and resistance over given periods of time. In the above expression R_o and A are unknown. What are known (or can be found experimentally) are the values of R_t at different times. For example, field data on terminal temperatures and flow rates taken on two different days would yield R_1 and R_2 at time t^1 and t_2 where $t_2 > t_1$ and $R_2 > R_1$. This gives us the following relationships:

$$R_1 = R_o (1 - e^{-At1}) \text{ or } R_o = R_1 / (1 - e^{-At1})$$

$$R_2 = R_o (1 - e^{-At2})$$

Combining these two expressions gives:

$$R_2/R_1 (1 - e^{-At1}) = 1 - e^{-At2}$$

We introduce the following:

$$1 - \alpha = e^{-At2} - \alpha e^{-At1},$$

where $\alpha = R_2/R_1$

Whereupon, the final working relationship is obtained:

$$(1 - \alpha) - e^{-At2} + \alpha e^{-At1} = 0$$

The working expression can be solved by trial and error to obtain a value for A. Once parameter A is known, the preceding expression can be used to calculate R_o. The input parameters needed for the analysis are R_1, R_2, t_1, and t_2.

CALCULATIONS FOR PIPE FLOWS

Estimating Pressure Drop for Turbulent Flow

Pressure drop calculations for simple geometries like pipe flows are straightforward. The Fanning friction factor is given by:

$$f = 0.054/Re^{0.2}$$

where: f = Fanning friction factor
        Re = Reynolds number

This correlation is good for a Reynolds number range of between 2,100 and 10^6. The Fanning equation for pressure drop is:

$$\Delta P_f = 2fLV^2\rho/g_cD$$

where: ΔP_f = Frictional pressure drop, lb/ft^2
        L = Pipe length, ft
        V = Velocity of fluid, ft/sec
        D = Pipe internal diameter, ft
        ρ = Fluid density, lb/ft^3
        gc = Gravitational constant, 32.2 lb$_m$-ft/lb$_f$-sec^2

Combining both expressions gives a simple relation for estimating the frictional pressure drop in straight run pipe.

$$\Delta P_f = 2\ [(0.054)/Re^{0.2}]\ [LV^2\rho\ /\ g_cD]$$

Determining Economic Pipe Diameter

The following analysis can be used to determine economic pipe diameters for the turbulent flow of Newtonian fluids. The working expression that can be used is:

$D^{4.84+n}\ /\ [1\ +\ 0.794L_cD]\ =$
$\{0.000189\ YKq^{2.8}\rho^{0.84}\mu^{0.16}((1\ +\ M)\ (1\ -\ \Phi)\ +\ ZM/(a'\ +\ b'))\}/\{n\times E(1\ +\ F)[Z\ +$
$(a\ +\ b)\ (1\ -\ \Phi)]\}$

The above expression can be solved by a trial and error calculation, whereby the following key assumptions are applied: (1) the flow is turbulent, i.e., Re $\geq$ 2,100, and (2) motive power is supplied by a prime mover such as a pump or a compressor. To make this expression easier to use, the terms are rearranged in the following manner:

$$P(D) = 0$$

where: $P = D^m - R - RSD$
$\qquad$ $m = 4.84 + n$
$\qquad$ $S = 0.794L_e$
$\qquad$ R = Right Hand Side of the Working Equation

The symbols in the working equation are defined as follows:

$\qquad$ D = Economical pipe diameter, ft
$\qquad$ L_e = Factor for friction in fittings, equivalent length in pipe diameter per unit length of pipe (1/ft)
$\qquad$ n = Exponent in pipe cost equation, $C = XD^n$; Note that C = Cost of pipe, $/ft; X = Cost of 1 ft of 1 ft diameter pipe, $/ft
$\qquad$ $M = EP(a' + b')/17.9 KY$
$\qquad$ where: E = Combined fractional efficiency of pump and motor
$\qquad\qquad\qquad$ P = Installed cost of pump and motor, $/hp
$\qquad\qquad\qquad$ K = Cost of power delivered to the motor, $/kW-hr
$\qquad\qquad\qquad$ Y = Days of operation per year
$\qquad$ a = Fractional annual depreciation on pipe line (dimensionless)
$\qquad$ b = Fractional annual maintenance on pipe line (dimensionless)
$\qquad$ Z = Fractional annual rate of return on incremental investment
$\qquad$ q = Flow rate, ft^3/sec
$\qquad$ F = Ratio of cost of fittings plus installation cost of pipe and fittings to cost of pipe only
$\qquad$ a' = Fractional annual depreciation on pumping installation
$\qquad$ b' = Fractional annual maintenance on pumping installation
$\qquad$ ϕ = Factor accounting for taxes
$\qquad$ μ = Fluid viscosity, cp
$\qquad$ ρ = Fluid density, lb/ft^3

Estimating Gas Temperatures in Pipelines

Gas temperature is an important quantity when designing gas transmission lines, since pipelines are designed for large flows and their consequent higher compressor requirements. The compressor power required at each station depends to a great extent on the gas temperature. Flowing gas temperature must be controlled to a

certain degree, particularly in very cold (Arctic) and very hot (Sahara) regions. The following analysis from the literature enables a estimating method for determining gas temperatures in pipelines. The steady state flow of natural gas in pipelihes obeys the first law of thermodynamics for a compressible fluid; which can be simply stated as follows:

$$m(dv/dx) + g(dz/dx) + (dh/dx) + (dq/dx) + (dw/dx) = 0$$

The enthalpy term h includes both internal energy and flow work terms. Since dw=0 between compressor stations, the equation becomes:

$$m(dv/dx) + g(dz/dx) + (dh/dx) = (dq/dx)$$

Neglecting changes in kinetic energy and potential energy produces:

$$dh/dx = dq/dx$$

For a compressible fluid that undergoes exansion through a valve or an orifice, the Joule-Thompson coefficient is defined as:

$$\mu = (\delta T/\delta P)_h$$

The relationship between heat content, temperature and pressure is defined by the following:

$$
\begin{aligned}
dh &= (\delta T/\delta P)_p dT + (\delta T/\delta P)_T dP \\
&= (\delta T/\delta P)_p dT + (\delta T/\delta P)_h \, (\delta T/\delta P)_p dP \\
&= (\delta T/\delta P)_p dT + \mu C_p dP \\
&= C_p dT + \mu C_p dP
\end{aligned}
$$

The conductive heat flow to the pipeline from the surrounding solid is:

$$dq/dx = (2\Pi UR/m) \, (T_g - T)$$

And combining the above expressions we obtain:

$$C_p \, (dT/dx) + \mu C_p \, (dP/dx) = (2\Pi UR/m) \, (T_g - T)$$

Defining $n = (2\Pi UR)/(mC_p)$, and introducing n into the above expression, we obtain an expression that after some simplification and rearrangement of terms that is based on assuming n and dP/dx to be constants:

$$dT/dx = -n \, (T - T_g) - \mu(dP/dx) = -n[T - T_g + (\mu/n)(dP/dx)]$$

This expression can be solved to provide:

$$\ln[T - T_g + (\mu/n)(dP/dx)] = -nx + C_1$$

The following boundary condition is applied: $x = 0$, $T = T_1$; and the solution is simplified to the following:

$$T = (T_1 - T_g + b)e^{-nx} + T_g - b$$

This last expression represents the working equation for the analysis, in which the terms are defined as follows:

b = Parameter defined as $(\mu/n)(dP/Dx)$
C_p = Specific heat of gas, Btu/lb-°F
g = Gravitational constant, 4.17×10^8 (lb_m/lb_f)(ft/hr^2)
h = Molal flow rate of gas, lb/hr
m = Mass flow rate of gas, lb/hr
n = Parameter defined as $2\delta UR/m \, C_p$
P = Pressure of gas at any section, psi
q = Heat flow to gas by conduction, convection and radiation, Btu/hr-lb
R = Radius of pipeline, ft
T_x = Temperature of the flowing gas at any section x-ft from the reference point, °F
T_1 = Temperature of the gas at the point of origin, °F
W = Thermodynamic flow work term
U = Heat transfer coefficient for transfer between the pipeline and surrounding ground, Btu/hr-ft^2-°F
X = Distance from origin, ft
Z = Distance above datum, ft
μ = Joule-Thompson coefficient
δ = Incremental amount of a variable

Pipeline Diameter Calculation for Compressible Gas Flows

The following analysis enables one to calculate the diameter of a pipeline transporting any compressible fluid. The required inputs are volumetric flow rate, the specific gravity of the gas relative to air, flow conditions, compressibility factor Z where Z is defined by nZRT = PV, the pressure at the point of origin and the destination, the pipe length, and pipe constants such as effective roughness. The working equations have been obtained from the literature. Since the friction factor

is an exponential function of the flow and therefore, the diameter, equations relating diameter, pressure drop, and flow are complex. The calculation procedure involves a trial-and-error method to compute the internal diameter, with the working equation being:

$$Q_b = 38.77 \; (T_b/P_b) \; [(1/G) \; (1/T_f) \; (1/Z)]^{1/2} D^{2.5} [(P_1^2 - P_2^2)/L]^{1/2} \; (1/f)^{1/2}$$

where: Q_b = Flow, scfd at base conditions
$\quad\quad$ T_b = Base temperature, 520 °R
$\quad\quad$ P_b = Pressure base, 14.73 psia
$\quad\quad$ G = Specific gravity of gas, relative to that of air = 1
$\quad\quad$ T_f = Flowing temperature, average °R
$\quad\quad$ Z = Supercompressibility, average for pipeline segment or at
$\quad\quad$ $P_{avg} = 2/3 \; [P_1 + P_2 - P_1 P_2/(P_1 + P_2)]$
$\quad\quad$ P_1 = Inlet pressure, psia
$\quad\quad$ P_2 = Outlet pressure, psia
$\quad\quad$ D = Inside pipe diameter, inches
$\quad\quad$ L = Segment length, miles
$\quad\quad$ f = Friction factor, dimensionless

Friction factor f is defined by $(1/f)^{1/2} = 4 \log (3.7 \; D/K_e)$, and K_e is equivalent pipe roughness, inches. The working equation may be simplified to the following:

$$Q_b = MD^{2.5} \log (3.7 \; D/K_e)$$

where: $M = 5476 \; (G \; T_f \; Z)^{-1/2} \; [(P_1^2 - P_2^2)/L]^{1/2}$

In performing computations, values should be tabulated as quantities of Q_b/M and $D^{2.5} \log(3.7D/K_e)$. If the difference in the two values is large, then a new guess of the diameter D is needed, or the value of the iteration increment must be changed.

Estimating Friction Factors

Determination of friction factors for some fluid flow applications can involves a trial-and-error procedure because the friction factor is not a simple function of the Reynolds number. Process engineers, therefore, refer to a Moody chart that has been developed using the following relationships:

$$f = 64/Re, \text{ for } 1 \le Re \le 2100, \text{ and}$$
$$1/\sqrt{f} = -2 \log \; [2.51/Re\sqrt{f} + \epsilon/3.7D], \text{ for } 2100 \le Re \le 10^6$$

In theses equations f is the friction factor, Re the Reynolds number, and ϵ/D is the relative roughness of the conduit. Inspection of the second equation reveals that for

turbulent flow, calculation of the friction factor involves a trial-and-error procedure. To avoid this method, another expression may be used to calculate the friction factor in all three flow regimes: laminar, transition, and turbulent. This expression is accurate for most engineering applications. The full-range friction factor is given as:

$$f = [(8/Re)^{12} + 1/(A + B)^{1.5}]^{1/12}$$

where parameters A and B are defined as follows:

$$A = [2.457 \ln\{1/\{(7/Re)^{0.9} + 0.27 \epsilon/D\}\}]^{16}$$

$$B = [37,530/Re]^{16}$$

Hence, to determine the friction factor, the input information needed is the relative roughness ϵ/D and the Reynolds number.

Petroleum Liquid Flows in Pipelines

The flow of petroleum liquids may be analyzed the same way as any other fluid flow problem. However, due to some industry convention, it is necessary to formulate an exclusive set of working equations. Examples of the "traditional ways" are expressing specific gravity in API units, viscosity in centistokes, flow in barrels per day, etc. Regular algorithms for fluid flow problems could very well be used, but the process engineer would have to convert the units before the making calculations. For this reason, the following estimation method has been designed to accept inputs in units commonly used in the petroleum industry. The working equations have been taken from the available.

$$H/L = C_1 Q^2/F^2 D^5$$

where: H = Total head loss, ft or meters
         L = Total pipe length, miles or km
         Q = Volumetric flow rate, bbl/hr or m³/hr
         D = Internal pipe diameter, inches or meters
         F = Fanning friction factor
         C_1 = Constant = 322.04, U.S. Customary System (USCS)
         = 25.508 x 10^{-6}, International System (SI) units

The value of C3 is 0.011454 in USCS units and 20.178 x 10^{-6} in S1 units. The inputs for the calculation are Q (bbl/hr or m³/hr) and pipeline length (miles or km), viscosity U (Centistokes), pipe diameter D (inches or meters), effective pipe roughness e, and pipeline lengths (miles or km). The Fanning friction factor is

related to the Darcy friction factor by definition:

$$F^2 = 4/f_D$$

Note that the Reynolds number is defined as follows:

$$Re = C2Q/D\mu$$

Where μ is defined as the kinematic viscosity (centistokes), and C_2 is a constant with a value of 2,213.8 in USCS units and 353.68 in SI units. An empirical relation for the Fanning friction factor is the Colebrook-White equation:

$$F = -4 \log (e/3.7D + 1.413 \, F/R)$$

By combining the above working expressions we obtain the final working expression for the analysis:

$$H/L = (C_1 Q^2/D^5) \, [-4 \log \{(e/3.7D) + (C_3\mu/D^{3/2})(1/(H/L))^{1/2}\}]^{-2}$$

And the constant C_3 is defined as follows:

$$C3 = 1.413 \, C_1^{1/2} / C_2$$

RECOVERY IN MULTICOMPONENT DISTILLATION

The following is a simplified estimating procedure for recovery in multicompnent distillation. In the working expressions provided below, the parameters b, d, and f rpresent the bottoms, distillate, and feed, respectively. Subscripts i, HK, and LK represent the component i, the heavy-key component, and the light-key component, repsectively. Relative volatility is represented by symbol α. Calculations can be readily set up on an Excel Spreadsheet.

$$d_i/f_i = 10^A \, \alpha_i^B / (1 + 10^A \, \alpha_i^B)$$

$$b_i/f_i = 1 / (1 + 10^A \, \alpha_i^B)$$

$$A = -\log [(b_{HK}/f_{HK})/(1 - b_{HK}/f_{HK})]$$

$$B = \log [\{(d_{LK}/f_{LK})/(1 - d_{LK}/f_{LK})\}\{(b_{HK}/f_{HK})/(1 - b_{HK}/f_{HK})\}] / \log (\alpha_{LK})$$

Physical properties data can be obtained from standard references such as the *CRC Handbook of Chemistry and Physics*.

ESTIMATING EQUILIBRIUM CURVES

In distillation work for binary systems with constant relative volatility, the equilibrium between phases for a given component can be expressed by the following equation:

$$y = \alpha x / \{1 - (1 - \alpha)x\}$$

where: x = Mole fraction in liquid phase

y = Mole fraction in vapor phase

α = Relative volatility

The procedure to calculate the points for an equilibrium curve is to start with $x = 0$, and calculate the value of y for a given value of α. A second point is calculated by incrementing the value of x and repeating the calculation for y. This calculation is repeated until the value of $x = 1$.

ESTIMATING EVAPORATION LOSSES FROM LIQUIFIED GASES

Liquified gases are sometimes stored in well-insulated spherical containers that are vented to the atmosphere. Examples in the industry are the storage of liquid oxygen and liquid ammonia in spheres. If the radii of the inner and outer walls are r_o and r_1, and the temperatures at these sections are T_o and T_1, an expression for the steady-state heat loss from the walls of the container may be obtained. A key assumption is that the thermal conductivity of the insulation varies linearly with the temperature according to the relation:

$$K = K_o + (K_1 - K_o) [(T - T_o) / (T_1 - T_o)]$$

The expression for the steady-state heat loss through the walls of the vessel is:

$$Q = 4\pi r_o r_1 [(K_o + K_1)/2][(T_1 - T_o)/(r_1 - r_o)]$$

The inputs required for the calculation are the radii, inner and outer temperatures, and thermal conductivities at the two temperatures. This expression enables an estimate of the heat flow into a spherical storage tank containing liquified gas.

COMBUSTION AIR CALCULATIONS

The following provides a calculation method for determining the amount of air needed for perfect combustion of one cubic foot of any gaseous fuel. The following expression provides an estimate of the ratio of the volume of air needed to the volume of fuel (i.e., the air to fuel ratio, Θ):

$\Theta = [(\%CH_4 \times 0.0956) + (\%C2H_6 \times 0.1673) + (\%C_3H_8 \times 0.239) + (\%C_4H_{10} \times 0.311) + (\%H_2 \times 0.0239) + (\%CO \times 0.0239) - \%O_2 \times 0.0478] \times [1 + XSA/100]$

In this equation all percentages are on a volume basis. The term XSA refers to the excess air over the stoichiometric requirement. The volumes of the air and gas must be measured at the same temperature and pressure. For consistency, it is best to first convert to actual conditions (i.e., from actual cubic feet (ACF) to standard cubic feet (SCF). The following formula can be used for this conversion:

$$SCF = (ACF) (36.05) (P_1 + 14.7) / (t_1 + 460)$$

The ACF is the actual cubic feet of gas measured at t_1 °F and P_1 psig. SCF represents standard conditions at 70 °F and 14.6 psia. The formulas provided require input information on the pressure and temperature of the fuel gas, the fuel gas analysis by volume (or mole percent if the pressures are sufficiently low), and the percent excess air. The calculation provides the air to fuel ratio required for complete combustion.

ESTIMATING TEMPERATURE PROFILES IN AGITATED TANKS

A common process task involves heating a slurry by pumping it through a well-stirred tank. It is useful to know the temperature profile of the slurry in the agitated vessel. This information can be used to optimize the heat transfer process by performing simple sensitivity studies with the formulas presented below. Defining the inlet temperature of the slurry as T_i, and the temperature of the outer surface of the steam coil as T_s; then by a macroscopic mass and energy balance for the system, a simplified calculation method is developed.

The system configuration is shown in Figure 7. The following system parameters are defined:

V = Volume of slurry in tank, ft^3
ρ = Density of slurry, lb$_m$/ft^3
W = Mass flow rate of slurry through tank, lb/hr
C_p = Heat capacity of slurry, Btu/(lb$_m$) (°F)
U = Overall heat-transfer coefficient of heating coil Btu (ft^2) (°F) (hr)
A = Total heat-transfer surface of coil, ft^2
t = Time since start of heating
$T(t)$ = Temperature of slurry in tank

Perfect mixing is assumed. The temperature profile of the tank with respect to time is then given by the following expression reported in the literature.

$$(T-T_\infty) / (T_i - T_\infty) = \exp[-\{(UA/\rho VC_p) + W/\rho V\}t]$$
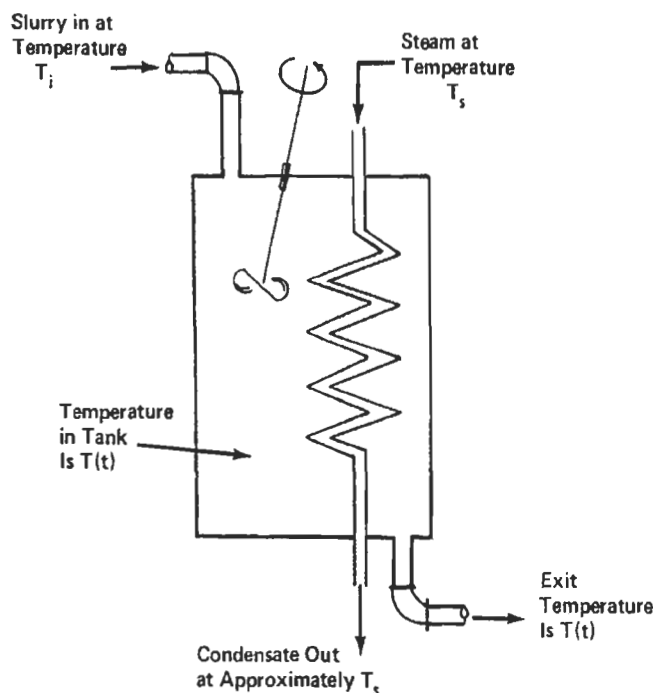
Figure 7. *System diagram for heated mixing tank.*

where:

$$T_{\infty} = [(UA/WC_p)T_s + T_i]/[1 + (UA/WC_p)]$$

By computing values from the above equations, a curve of T versus time may be plotted. This information may be used to design heating systems or evaluate the performance of existing ones. The input parameters required for the analysis are T_i, U, A, W, C_P, and T_s.

GENERALIZED EQUATIONS FOR COMPRESSORS

A process engineer's task is often to evaluate the performance of a compressor unit based on gas throughputs and terminal pressures. Since compressor stations are complex machines and operations, the analysis required is sophisticated and goes well beyond simple computations on a personal computer, although some preliminary evaluations can certainly be made. In this section we summarize the working expressions for standard compressor operations. Compressor operations can be categorized under three thermodynamic categories:

1. *Isothermal.* When compression takes place at constant temperature. This situation

is strictly hypothetical. A constant-temperature operation can be approached only when the compressor runs at an infinitely slow speed.

2. *Adiabatic.* When the net heat lost or gained by the unit to or from the surroundings is zero. Most plant installations approach this operation, and the adiabatic equations are widely used.

3. *Polytropic.* Sometimes the compression process has certain associated irreversibilities. The actual operation is therefore approaching adiabatic, but not quite. This "approximately adiabatic" operation is called polytropic.

In evaluating and/or designing compressors the main quantities that need to be calculated are the outlet (discharge) gas temperature, and the energy required to drive the motor or other prime mover. The latter is then corrected for the various efficiencies in the system. The differential equations for changes of state of any fluid in terms of the common independent variable are derived from the first two laws of thermodynamics:

$$dQ = TdS = C_v dT + T \, (\partial P/\partial V)_v dV$$

$$dQ = TdS = C_p dT - T(\partial V/\partial T)_p dP$$

$$W_{cycle} = -\int VdP,$$

where Wcycle refers to Cycle Work, and the integration is over the limits of P_1 to P_2. In the case of isothermal compression, for an ideal gas, we may state the following:

$$W = \int PdV = RT \int dv/v = - RT \int dP/P, \text{ or}$$

$$W = - RT \ln [P_2/P_1]$$

The above expression is valid for either compression or expansion between the given pressure limits. For the case of compression, $P_2 > P_1$, and the work is negative (i.e., work is done on the system). For expansion, $P_2 < P_1$, and the work term is positive (i.e., work is performed by the system). If compression is isothermal and the gas is ideal, then the cycle work per mole is also given by the last equation stated above.For adiabatic compression, the adiabatic change is described by the following equations, where k is the ratio of specific heats and some typical values for common gases are : 1.67 for monatomic gases (e.g., He, A, etc.); 1.40 for diatomic gases (e.g., H_2, CO, N_2); and 1.30 for tri-, tetra- and penta-atomic gases (e.g., CO_2, CH_4, etc.):

$$P_1 V_1^k = P_2 V_2^k = P_3 V_3^k = ,...$$

$$(T_2/T_1) = (P_2/P_1)^{(k-1)/k}$$

$$W_{Theoretical} = \{RT_1/(k-1)\}[1 - (P_2/P_1)^{(k-1)/k}]$$

$$W_{Cycle} = (NK)(W_{Theoretical})$$

For polytropic compression, the change may be described by the following equations:

$$P_1V_1^n = P_2V_2^n = P_3V_3^n = ,...$$

$$(T_2/T_1) = (P_2/P_1)^{(n-1)/n}$$

$$W_{Theoretical} = \{RT_1/(n-1)\}[1 - (P_2/P_1)^{(n-1)/n}]$$

$$W_{Cycle} = (Nn)(W_{Theoretical})$$

Since non-ideal gases do not obey the ideal gas law (i.e., $PV = nRT$), corrections for nonideality must be made using an equation of state such as the Van der Waals or Redlich-Kwong equations. This process involves complex analytical expressions. Another method for a nonideal gas situation is the use of the compressibility factor Z, where Z equals PV/nRT. Of the analytical methods available for calculation of Z, the most compact one is obtained from the Redlich-Kwong equation of state. The working equations are listed below:

$$h = b/V = (0.0867RT_c)/VP_c$$

$$Z = [1/(1 - h)] - (4.934/T_r^{1.5})[h/(1 + h)]$$

And form $PV = NZRT$, we note that $T = PV/NZR$, and hence, at any initial state 1, we may write the following:

$$T_1 = (P_1V_1)/(NZ_1R)$$

Combining the above expressions results in the following equation for the cycle work required in an adiabatic change:

$$W_{Cylce} = kV_1/\{N(k-1)Z_1\}[P_1 - (P_2/P_1)^{(k-1)/k}]$$

Similarly, the cycle work required in a polytropic process is given by the following expression:

$$W_{polytropic} = nV_1/\{N(n-1)Z_1\}[P_1 - (P_2/P_1)^{(n-1)/n}]$$

For an isothermal change, the expression for P from the Redlich-Kwong equation can be substituted into the general formula for work done:

$$W = \int P dV$$

$$P = (RT)/(V - b) - (a)/(T^{1/2}V(V + b))$$

Substituting and combining above expressions we obtain:

$$W = \int [\{RT/(V - b)\}dV - adV/(T^{1/2}V(V + b))]$$

The above integral can be evaluated either analytically or numerically by applying Simpson's rule. The following provides a summary of the major working equations for compressor analysis:

For the discharge temperature in an adiabatic process:

$$T_2 = T_1(P_2/P_1)^{(k-1)/k}$$

For the discharge temperature in a polytropic process:

$$T_2 = T_1(P_2/P_1)^{(n-1)/n}$$

For theoretical cycle work performed in an adiabatic compression cycle (nonideal fluid):

$$W = kV_1/\{N(k-1)Z_1\}[P1 - (P_2/P_1)^{(k-1)/k}];$$

$$Z = [1/(1 - h)] - (4.934/T_r^{1.5})[h/(1 + h)];$$

$$h = 0.0867 \, RT_c/VP_c$$

For the theoretical cycle work performed in a polytropic compression cycle (nonideal fluid):

$$W = nV_1/\{N(n-1)Z_1\}[P_1 - (P_2/P_1)^{(n-1)/n}]$$

For theoretical cycle work performed in an isothermal compression cycle:
(For ideal fluid case)
$$W = -RT \ln(P_2/P_1)$$
(For nonideal fluid case)
$$W = \int[\{RT/(V - b)\}dV - adV/(T^{1/2}V(V + b)]$$

BATCH DISTILLATION: APPLICATION OF THE RAYLEIGH EQUATION

In discontinuous simple open distillation (batch distillation, Rayleigh distillation), the distillation still is charged with a liquid mixture (feed). Heating to the boiling point partially vaporizes the liquid. The vapor is condensed and collected in the distillate receiver. Refer to Figure 8 for a simplified definition of the operation.
Batch distillation is a dynamic process. The composition of distillate and liquid remaining in the still (residue) as well as temperature change with time. Figure 9 illustrates these changes qualitatively. At any time during the distillation, the total number of moles in the still is n_R with mole fraction x_R of the considered component. After distilling an incremental amount dn_R with mole fraction y, the number of moles in the still is n_R - dn_R. We may now write a component material balance as follows:

$$ydn_R = n_R x_R - (n_R - dn_R)(x_R - dx_R)$$

Neglecting $dn_R dx_R$ as the product of two small quantities, the following differential equation is derived which relates the amount of residue in the still to the composition:

$$dn_R/n_R = dx_R / (y - x_R)$$
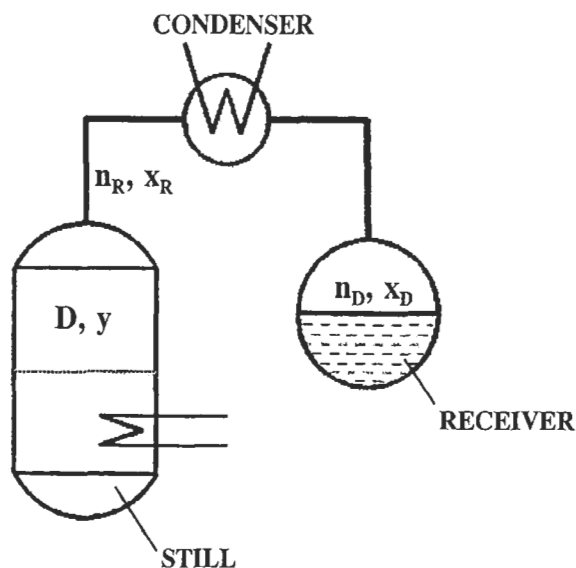
Figure 8. *Simple batch distillation.*
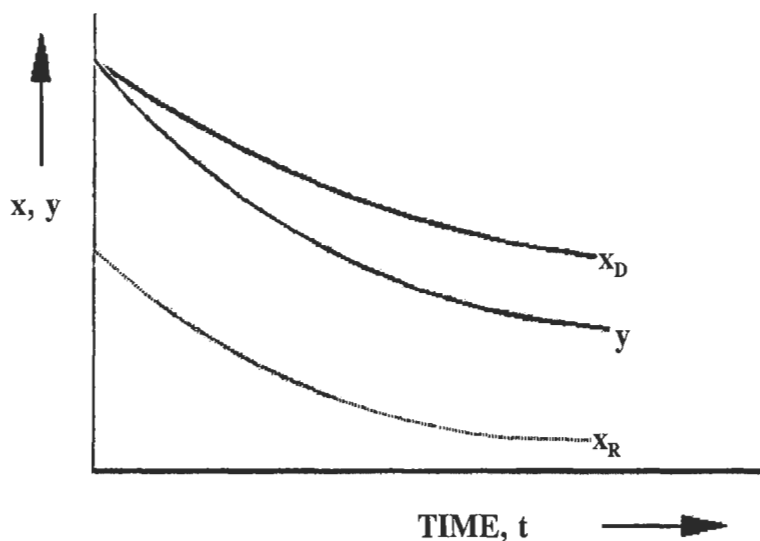
Figure 9. *Dynamic equilibrium curve for batch distillation.*

This equation was first given by Lord Rayleigh and is called the Rayleigh equation. Integration between the initial number of moles n_{R0} in the still with composition x_R over any time yields the following:

$$\ln (n_R/n_{R0}) = \int 1/(y - x_R) \, dx_R$$

Note that this equation holds for any component in a multi-component mixture. The integral on the right-hand side can only be evaluated if the vapor mole fraction y is known as a function of the mole fraction x_R in the still. Assuming phase equilibrium between liquid and vapor in the still, the vapor mole fraction $y(x_R)$ is defined by the equilibrium curve. Agitation of the liquid in the still and low boilup rates tend to improve the validity of this assumption.

By using vapor-liquid equilibrium data the above integral can be evaluated numerically. A graphical method is also possible, where a plot of $1/(y - x_R)$ versus x_R is prepared and the area under the curve over the limits between the initial and final mole fraction is determined. However, for special cases the integration can be done analytically. If pressure is constant, the temperature change in the still is small, and the vapor-liquid equilibrium values (K-values, defined as K=y/x for each component) are independent from composition, integration of the Rayleigh equation yields :

$$n_R/n_{R0} = [x_R/x_{R0}]^{1/(K-1)}$$

More often than the assumption of constant K-values, the assumption of constant relative volatilities is applied. The relative volatility of two components "i" and "j" is defined as the ratio of their K-values:

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

For a binary mixture with constant relative volatility the following expression applies:

$$\alpha = y(1 - x_R) / x_R(1 - y)$$

It then follows that:

$$\ln(n_R/n_{R0}) = \{1/(\alpha - 1)\}\{\ln(x_{R0}/x_R + \alpha \ln[(1 - x_R)/(1 - x_{R0})]\}$$

The total number of moles n_D and composition x_D in the distillate receiver can now be obtained from the material and component material balances:

$$n_D = n_{R0} - n_R$$

$$x_D = [n_{R0} x_{R0} - n_R x_R]/n_D$$

Assuming a specific boilup rate "D", the compositions may now be calculated as a function of time:

$$dx_R/(y - x_R) = -(D/n_R)dt$$

where

$$n_R = n_{R0} - \int D dt$$

The above integration is performed over the limits of 0 to time t. Again, assuming D to be a constant, then:

$$\int dx_R/(y - x_R) = \ln[1 - Dt/n_{R0}]$$

Upon integration, we obtain the final working expression which can be readily programmed on to a spreadsheet for calculations:

$$x_R/x_{R0} = \ln[1 - Dt/n_{R0}]$$

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