

INTRODUCTION

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Mixing as a discipline has evolved from foundations that were laid in the 1950s, culminating in the publication of works by Uhl and Gray (1966) and Nagata (1975). Over the last 30 years, many engineering design principles have been developed, and design of mixing equipment for a desired process objective has become possible. This handbook is a compilation of the experience and findings of those who have been most active in these developments. Together, the authors' experience extends over more than 1000 years of research, development, and consulting work.

This book is written for the practicing engineer who needs to both identify and solve mixing problems. In addition to a focus on industrial design and operation of mixing equipment, it contains summaries of the foundations on which these applications are based. To accomplish this, most chapters have paired an industrialist and an academic as coauthors. Discussions of theoretical background are necessarily concise, and applications contain many illustrative examples. To complement the discussions, a CD ROM is included which contains over 50 video clips and animations of mixing processes. These clips are accompanied by explanatory text. Internal cross-referencing and external references are used extensively to provide the reader with a comprehensive presentation of the core topics that constitute current mixing practice.

The core mixing design topics are:

- Homogeneous blending in tanks and in-line mixers
- Dispersion of gases in liquids with subsequent mass transfer
- Suspension and distribution of solids in liquids

- Liquid–liquid dispersions
- Heat transfer
- Reactions: both homogeneous and heterogeneous

Underlying principles are presented in chapters on:

- Residence time distribution
- Turbulence
- Laminar blending and flow

Additional information is provided on ways of investigating mixing performance:

- Experimental measurement techniques
- Computational fluid dynamics

These topics are augmented by chapters on specific industrial mixing topics:

- Solid–solid blending
- Polymer processing
- Fine chemical and pharmaceutical processes
- Fermentation and cell culture
- Petroleum
- Pulp and paper
- Mixing equipment: vessels, rotor–stators, and pipeline mixers
- Mechanical aspects of mixing equipment
- The vendor’s role

At the end of this introduction, a set of charts is provided for the initial assessment of mixing related problems. These charts are designed to assist the reader who is meeting a mixing problem for the first time, and is unsure of where to start. They are not meant to replace the senior engineer or mixing specialist, who will typically be able to quickly evaluate the key issues in mixing-sensitive processes.

MIXING IN PERSPECTIVE

What is mixing? We define *mixing* as the reduction of inhomogeneity in order to achieve a desired process result. The inhomogeneity can be one of concentration, phase, or temperature. Secondary effects, such as mass transfer, reaction, and product properties are usually the critical objectives.

What constitutes a mixing problem? Process objectives are critical to the successful manufacturing of a product. If the mixing scale-up fails to produce the

required product yield, quality, or physical attributes, the costs of manufacturing may be increased significantly, and perhaps more important, marketing of the product may be delayed or even canceled in view of the cost and time required to correct the mixing problem.

Although there are many industrial operations in which mixing requirements are readily scaled-up from established correlations, many operations require more thorough evaluation. In addition to presenting the state of the art on the traditional topics, this book presents methods for recognition of more complex problems and alternative mixing designs for critical applications.

Failure to provide the necessary mixing may result in severe manufacturing problems on scale-up, ranging from costly corrections in the plant to complete failure of a process. The costs associated with these problems are far greater than the cost of adequately evaluating and solving the mixing issues during process development. Conversely, the economic potential of improved mixing performance is substantial. Consider the following numbers:

- *Chemical industry.* In 1989, the cost of poor mixing was estimated at \$1 billion to \$10 billion in the U.S. chemical industry alone. In one large multinational chemical company, lost value due to poor mixing was estimated at \$100 million per year in 1993. Yield losses of 5% due to poor mixing are typical.
- *Pharmaceutical industry.* Three categories should be considered: costs due to lower yield (on the order of \$100 million); costs due to problems in scale-up and process development (on the order of \$500 million); and costs due to lost opportunity, where mixing problems prevent new products from ever reaching the market (a very large number).
- *Pulp and paper industry.* Following the introduction of medium consistency mixer technology in the 1980s, a CPPA survey documented chemical savings averaging 10 to 15% (Berry, 1990). Mills that took advantage of the improved mixing technology saw their capital investment returned in as little as three months.

From these numbers, the motivation for this handbook and for the research efforts that it documents becomes clear. The reader will almost certainly profit from the time invested in improved understanding of the design of mixing equipment. *Mixing equipment design must go beyond mechanical and costing considerations, with the primary consideration being how best to achieve the key mixing process objectives. Mixing solutions focus on critical issues in process performance.*

How much mixing is enough, and when could overmixing be damaging to yield or quality? These critical issues depend on the process and the sensitivity of selectivity, physical attributes, separations, and/or product stability to mixing intensity and time. The nonideality of residence time distribution effects combined with local mixing issues can have a profound effect on continuous processes.

Useful methods for mixing process development effort have been evolving in academic and industrial laboratories over the past several decades. They include improvements to traditional correlations as well as increasingly effective methods both for experiments and for simulation and modeling of complex operations. The combination of these approaches is providing industry with greatly improved tools for development of scalable operations. This handbook provides the reader with all the information required to evaluate and use these technologies effectively in process development and scale-up.

How should new mixing problems be solved? Solutions for new mixing problems require answers to the question “Why?” as well as the very pressing question “How?” This question is best addressed with a good understanding of both the process and the underlying fundamentals. This requires discussion with both operations and developmental chemists. It is often well served by reposing the question “How can we scale this up?” as “How can we scale down the process equipment to closely replicate plant conditions in the lab?” The importance of this question should never be underestimated, as it often opens the door for discussions of geometric similarity and matching of mixing conditions. Good experimental design based on an understanding of mixing mechanisms is critical to obtaining useful data and robust solutions. Engineers who ignore the fundamentals always do so at their own peril. It is our hope in writing this book that mixing fundamentals will become accessible to a much wider audience of engineers, chemists, and operators whose processes are affected by mixing issues.

Scope of Mixing Operations

Mixing plays a key role in a wide range of industries:

- Fine chemicals, agrichemicals, and pharmaceuticals
- Petrochemicals
- Biotechnology
- Polymer processing
- Paints and automotive finishes
- Cosmetics and consumer products
- Food
- Drinking water and wastewater treatment
- Pulp and paper
- Mineral processing

In all of these industries, the components of mixing problems can be reduced to some fundamental concepts and tools. The key variables to identify in any mixing problem are the time available to accomplish mixing (the time scale) and

the required scale of homogeneity (the length scale of mixing). In the remainder of this section we briefly summarize the key mixing issues, the time and length scales of interest, from the perspective of key mixing objectives. We begin with residence time distributions, since this is typically the only area of mixing covered in the undergraduate curriculum.

Residence Time Distributions: Chapter 1

Classical reactor analysis and design usually assume one of two idealized flow patterns: plug flow or completely backmixed flow. Real reactors may approach one of these; however, it is often the nonidealities and their interaction with chemical kinetics that lead to poor reactor design and performance (Levenspiel, 1998). Nonidealities include channeling, bypassing, and dead zones, among others.

A well-known method for assessing the nonideality of continuous process equipment is the determination of fluid residence time distributions. Residence time distribution (RTD) is a concept first developed by Danckwerts in his classic 1953 paper. In RTD analysis, a tracer is injected into the flow and the concentration of tracer in the outlet line is recorded over time (see Chapter 4). From the concentration history, the distribution of fluid residence times in the vessel can be extracted.

The limits of RTD analysis are the ideal plug flow of a pulse of tracer and a perfectly mixed pulse of tracer. In plug flow a pulse that is completely isolated from the rest of the reactor volume travels through the vessel in exactly the mean residence time. In a perfectly mixed stirred tank, the pulse of tracer is immediately mixed with the full volume of the reactor, leaving the vessel with an exponential decay of concentration as the volume is diluted with fresh feed. These two ideal limits provide us with a great deal of information about the bulk flow pattern or macromixing. When the mixing is ideal or close to ideal and the reaction kinetics are known, the RTD can be used to obtain explicit solutions for the reactor yield [see Levenspiel's classic introductory discussion (1972), Baldyga and Bourne's summary of the key cases (1999, Chap. 2), and Nauman's comprehensive treatment (2002)]. For many industrially important applications, the ideal and close-to-ideal models work very well.

The chief weakness of RTD analysis is that from the diagnostic perspective, an RTD study can identify whether the mixing is ideal or nonideal, but it is not able to uniquely determine the nature of the nonideality. Many different nonideal flow models can lead to exactly the same tracer response or RTD. The sequence in which a reacting fluid interacts with the nonideal zones in a reactor affects the conversion and yield for all reactions with other than first-order kinetics. This is one limitation of RTD analysis. Another limitation is that RTD analysis is based on the injection of a single tracer feed, whereas real reactors often employ the injection of multiple feed streams. In real reactors the mixing of separate feed streams can have a profound influence on the reaction. A third limitation is that RTD analysis is incapable of providing insight into the nature

of micromixing. RTD studies and analyses deal primarily with bulk flow or macroscopic mixing phenomena.

Where do the ideal models fail? For flow in a pipe, the ideal model is plug flow. This is a good assumption for fully turbulent flow with a uniform distribution of feed. There are two important cases where nonideal mixing must be addressed. If the second component is added from a small feed pipe rather than as a slug, radial dispersion of the feed must be considered. This case is discussed in Chapter 7. If the flow is laminar rather than turbulent, the velocity profile is parabolic (not flat), so the fluid in the center of the pipe will exit much sooner than the fluid close to the walls. This is the laminar axial dispersion problem which has been studied very extensively. The animation of flow in a Kenics mixer (CD ROM) illustrates this concept, showing axial dispersion of tracer particles for laminar flow in a static mixer. Ways to avoid this problem are also discussed in Chapter 7. For turbulent flow the problem of axial dispersion is less severe. A third practical consideration is partial plugging or fouling of a line. In this case the apparent residence time will be much shorter than expected because the effective volume of the vessel is less than the design volume.

For well-designed stirred tanks with simple reaction schemes and kinetics which are slow relative to the mixing time, the perfectly backmixed CSTR model works well. The most critical factor for design of a CSTR is placement of the feed and outlet locations. If a line drawn from the feed pipe to the outlet passes through the impeller, short circuiting is not likely to be a problem. If, however, the feed and the outlet are both located near the top of the vessel, short circuiting will almost certainly occur. Baffles may be used to reduce or eliminate this problem. The second characteristic of a well-designed CSTR is that the volume and mixing must be balanced with the feed rate. The volume must be big enough to allow 10 batch blend times to occur over the mean residence time (see Chapter 6). Alternatively, the primary impeller pumping capacity (see Chapter 6) should be 10 to 16 times the volumetric feed rate $q/Q = 10$ to 16 (Nauman, 2002, Chap. 8). These numbers are very conservative but are the best design standards currently available.

Residence time distributions, discussed in Chapter 1, represent the first generation of mixing models. The ideal cases of plug flow and perfectly mixed tanks provide solutions for most standard problems. Where the kinetics are more complex, are faster than the mixing time, or require a segregated feed strategy, the local mixing concepts discussed in this book and the zone-based models developed over the last 20 years have proved invaluable. The third generation of modeling will see coupling of computational fluid dynamics (Chapter 5) with reaction kinetics and heat transfer to obtain explicit and localized models for the most difficult mixing problems. Early reports of successes in this area include the production of adipic acid in the laminar flow regime in a stirred tank, modeling of crystallization reactions, and evaluation of the disinfection capabilities of ultraviolet treatment reactors in the water and wastewater treatment industries.

Residence time distributions are the first characteristic of mixing, but because they treat the vessel as a black box, they cannot address local mixing issues, which are the focus of much of this book. The characteristic time scale for a residence time distribution is the mean residence time of the vessel. The characteristic length scale is the vessel diameter, or volume. Many of the key process objectives of interest require more local information.

Mixing Fundamentals: Chapters 1–5

There is a set of fundamental topics which, while not leading directly to design of mixing equipment, must be understood to address difficult mixing problems. Residence time distribution theory and modeling constitute the classical approach to mixing and were discussed earlier. Turbulent and laminar mixing theory is covered in Chapters 2 and 3. Laminar mixing theory springs from dynamical systems theory, or chaos theory. A number of topics are addressed, but perhaps most useful is the idea that well-designed laminar mixing devices repeat the stretching and folding patterns in the flow, thus producing repeating structures of mixing on ever smaller scales. Turbulent mixing theory is concerned primarily with two questions: “What is the range of time and length scales in the flow?” and the analog to this question, “Where is the energy dissipated?” The points of highest energy dissipation are the points of most intense mixing, or of the smallest time and length scales. Chapters 4 and 5 discuss the two principal tools used to investigate mixing phenomena and evaluate mixing equipment: laboratory experiments and computational fluid dynamics. There is a wide range of experimental and computational tools available with a wide range of experimental or computational difficulty and a wide range of detail in the results. Perhaps the most difficult question for the engineer is to understand the problem well enough to define a well-posed question. Once the question is defined, an appropriate tool can be selected relatively easily, and useful results can usually be obtained. These five fundamental topics provide the key tools needed to tackle new problems and to understand much of the theory underlying mixing design.

Mixing Equipment: Chapters 6, 7, 8, and 21

A wide range of mixing equipment is now available, with the current generation of equipment typically designed for a specific process result. Chapter 6 covers traditional stirred tanks, baffling, the full range of impellers, and other tank internals and configurations. Chapter 7 provides information on equipment and design for pipeline mixing. Chapter 8 focuses on rotor–stators, which have been used for many years but have been investigated on a more fundamental level only in the last decade. Chapter 21 covers the mechanical aspects of mixing equipment design, providing a welcome primer on the vocabulary of mechanical engineering as well as important design information. Chapter 22 focuses on the vendor: what expertise can be offered and what information is needed for accurate specification of mixing equipment. Additional specialized equipment is discussed in

Chapters 15 (powder blending), 16 (high viscosity), 19 (petroleum), and 20 (pulp and paper industry). Key design concepts for equipment selection are:

- Selection of tanks versus in-line mixers and use of backmixed flow versus plug flow
- Selection of residence times are required: long residence times are well served by tanks, short residence times can be accomplished in pipes
- Design requirements: robust and flexible (typically stirred tanks) versus tight and specific (pipeline mixers and other specialized equipment)
- Mechanical design considerations: seals, dynamic loads, rotating shafts, and critical speed
- Classical and modern impeller design; the function and importance of baffles
- Characteristics of in-line mixing equipment, including static mixers and rotor–stators

Miscible Liquid Blending: Chapters 3, 7, 9, and 16

Miscible liquid blending is the easiest mixing task. The reader is cautioned that miscible blending requires two things: The streams must be mutually soluble, and there must be no resistance to dissolution at the fluid interface. Chapters 7 and 9 present well-developed correlations for prediction of mixing time in this simplest case, and corrections for density and viscosity differences. Although laminar and non-Newtonian fluids are more difficult to handle, the current recommendations on these issues are also included in Chapters 7 and 9.

Chapter 3 provides a careful discussion of how we characterize and measure mixing scales. These concepts are combined with dynamical systems, or chaos theory, to identify similarities of scale in laminar mixing applications. This is a key theoretical concept that will allow rigorous advances in mixing design in the future. In Chapter 16, current polymer and high viscosity blending equipment is discussed. In these cases the blending objective must be combined with the heat transfer and high pressures required to produce polymer melts. For pastes, the fluids are typically non-Newtonian, so further specialized equipment is required.

Solid–Liquid Suspension: Chapters 10, 17, and 18

Design methods for solid–liquid suspension were some of the first to be established (Zwietering, 1958), and this early work has withstood the test of time virtually unchanged. Solid–liquid mixing is discussed in Chapter 10, with design guidelines for:

- Mixing requirements for achieving and maintaining off-bottom suspension of solids (the just suspended speed, N_{js})

- Requirements for achieving and maintaining uniform solids concentration throughout the tank—of interest particularly for slurry catalyst reactors and for feeding downstream equipment (e.g., centrifuges, continuous stirred tank reactors, fluid bed coaters)
- Mass transfer correlations for solids dissolution
- Maintaining the required slurry composition on discharge
- Tank draining with solids present: avoiding plugged nozzles

Difficult design problems that have not yet been resolved involve nonwetting, clumping, or floating solids. The key qualitative aspects of these problems can be identified and useful heuristic solutions are provided. Other mixing effects involving solids in suspension include clumping, agglomeration, fouling, and scaling. These problems can be reduced with good mixing designs, but a full discussion lies outside the scope of this book.

Reactions involving solids are discussed extensively in Chapters 13 and 17. Where solids are involved in reactions, there are two steps in the kinetics. The first, solids dissolution, is dominated by the particle size and the mixing conditions. The apparent reaction kinetics and even the reaction products can change depending on the mixing conditions. Key solids reaction topics include:

- Solids dissolution with reaction (Chapters 13 and 17)
- Potential for impeller damage to solids in suspension, including crystals (Chapter 17), cells (Chapter 18), and resin beads
- Mixing effects on nucleation and growth in crystallization (Chapter 17)

Gas–Liquid Contacting: Chapter 11

Gas–liquid mixing has one key objective: the dispersion of gas in liquid with the maximum surface area for mass transfer. As with many multiphase systems, this objective is complicated by the difficulties of multiphase flow. The gas can flood the impeller, dramatically reducing its effectiveness; surface properties determine whether the system is coalescing or noncoalescing, and thus whether the surface area created is stable; boiling systems require completely different treatment; and gas–liquid reactions require consideration of local concentrations of gas. Chapter 11 includes the traditional topics:

- Correlations for prediction of $k_L a$, including fermentation applications (also discussed in Chapter 18)
- Discussion of operating regimes: interaction of power and gassing rate to produce stable operation or flooding of the impeller
- Recommendations for sparger design and placement
- Design for sufficient gas phase residence time
- Gas–liquid reactions (also discussed in Chapter 13)

New discussions are provided on:

- The new generation of impellers designed for efficient gas dispersion
- Boiling systems

The reader should beware of conditions in the headspace, particularly for high viscosity and/or foaming systems. This is potentially detrimental for several types of operations. Excessive foaming can lead to interference with mass transfer. Gas entrained into high viscosity systems can be difficult to remove and severely affect product quality.

Liquid–Liquid Mixing: Chapter 12

Liquid–liquid mixing is one of the most difficult and least understood mixing problems, despite extensive literature on both the mechanical agitation side of the problem and the surface science side of the problem. In spite of this, a number of important lessons emerge from the discussion in Chapter 12:

- Impurities, surface-active agents, and small changes in chemical composition can be critical in determining drop size distribution. Performance can change dramatically due to small changes in composition, even at the parts per million level, particularly for reactions, separations, and preparation of stable emulsions.
- Both the mixing system and duration of mixing can have an important effect on drop size distribution, drop breakup, and coalescence.
- Addition strategy can determine which phase is continuous.
- Phase inversion can play an important role in extraction and reaction.
- Overmixing can result in a stable emulsion or an overreacted product.
- Inadequate mixing can result in incomplete phase transfer or slow reaction.

Mixing and Chemical Reactions/Reactor Design: Chapters 13 and 17

When mixing rates and chemical reaction rates occur on similar time scales, or when mixing is slower than chemical reaction, mixing effects can be very important. On the small scale, blend times and mixing time scales are typically very short and mixing effects may not be apparent. When reactions are scaled up, however, the chemical kinetics stay the same while mixing times get longer. Mixing effects are always worse on scale-up. These issues are discussed in some detail in Chapters 13 and 17. The key points are:

- How and when mixing effects can influence the yield and selectivity of complex homogeneous and heterogeneous chemical reactions.

- Yield and/or selectivity of homogeneous consecutive-competitive reactions that are subject to mixing effects can be lower on scale-up if proper precautions are not taken for mixing the reagents—mesomixing problems get worse on scale-up and blend times increase.
- Feeding at the impeller, or the region of most intense turbulence, is recommended for consecutive or competitive reactions to avoid reduced yield/selectivity on scale-up. It is better to feed at the impeller when this is not actually required than to feed on the surface when subsurface feed was in fact necessary.
- Mixing effects in heterogeneous reactions are often complex because of local effects in dispersed phase films and global mixing effects when competitive reaction(s) occur in the continuous phase.
- The yield and/or selectivity of heterogeneous complex reactions may in some cases be improved by the presence of the second or third phase.

Heat Transfer and Mixing: Chapter 14

The principles of heat transfer in stirred tanks are discussed in Chapter 14, with a full set of design correlations for heat transfer coefficients. The key heat transfer concepts to keep in mind are as follows:

- Limitations in heat transfer normally result from surface area availability rather than from the mixing system.
- Limitations in heat transfer can sometimes be overcome by evaporative cooling: for example, during polymerization and other exothermic reactions.
- Good mixing can often reduce or prevent scaling and the resulting severe losses in heat transfer performance.
- Process modifications are sometimes needed to provide alternative solutions to limitations in heat transfer capability.

Specialized Topics for Various Industries: Chapters 15–20

Mixing issues in several specialized industries are discussed in Chapters 15 to 20. In these chapters, the approach taken varies from author to author, depending on the state of knowledge in the industry. Powder blending and polymer or high viscosity blending both suffer from the difficulty of even characterizing the material of interest, making fully predictive design and scale-up nearly impossible. The fine chemicals industry typically uses equipment for a wide range of products, so the mixing must be both versatile and well understood. Reactions are often multiphase, and crystallization is a core competency with its own specialized mixing issues. Chapter 17 can be regarded as a more specialized reactions chapter. Biological processes, discussed in Chapter 18, are highly dependent on gas dispersion but must also consider the special requirements of living systems. The petroleum and pulp and paper industries have a range of key applications.

These applications are the focus of Chapters 19 and 20 and have significant value for extending one's understanding of mixing design fundamentals.

CONVERSATIONS OVERHEARD IN A CHEMICAL PLANT

One Monday morning in an R&D center in Illinois, Marco's phone rings. "Hello—Marco? It's Bill from the Texas plant." Marco detects a mixture of excitement and concern in Bill's voice, "What's up, Bill?" "Well—ah—Marco, remember that mixing vessel you designed for us? Listen, we moved the reaction in Step 5 of the process—you remember the liquid–liquid phase transfer reaction step—to another vessel. The conversion and selectivity are both lower. We have checked the usual suspects, compound III from the previous step, temperature calibration, and charge meter calibrations, but everything looks OK. Any chance the change in vessel could be giving us a mixing problem?" Marco shakes his head and replies, "Very good chance, Bill. These liquid–liquid fast reactions can be devils to scale up. Let's get together and see what we can do."

In the chapters that follow, the varied roles of mixing in industrial operations are discussed by authors from both academic and industrial viewpoints, combining the fundamentals of mixing technology with industrial experience. Many examples are included, providing both illustrative calculations and more qualitative industrial mixing problems. In this section we follow Marco's journey as he works through the mixing issues that must be considered in development of a new process and its translation to manufacturing.

The Problem

Marco first heard about this project when his boss, the director of chemical engineering R&D, called with the news that a new process was coming out of research with good potential to go through development and into manufacturing as the company's next product. "Talk to the head chemist, Lenny, and find out what the process looks like. Determine what engineering issues it may involve on scale-up as well as potential areas for process improvement."

This type of assignment had come to Marco many times before and always caused him concern. Achieving a successful scale-up always requires development of multiple steps that are easy to operate in the laboratory but can be very difficult to translate to manufacturing. In many cases a direct scale-up of the chemists' procedure is possible, but in others, mixing differences between large- and small scale equipment result in reduced yield and selectivity. One of Marco's first objectives was to determine the scalability of the new process in each of the reaction, purification, and isolation steps. In order to focus on the mixing issues of interest, the aspects of this process that require feasibility and optimization studies are not addressed in this discussion.

On initial review of the chemists' procedure, Marco notes that there are potential mixing issues in four of the chemical reactions as well as in the crystallization

steps. The other steps in the process did not appear to pose significant mixing issues beyond prudent scale-up of blending, solids suspension, and so on. This initial diagnosis is critical to the success of the development program that Marco will put together in consultation with his colleagues. Marco is well aware that mixing problems can be difficult to forecast from a laboratory procedure and that careful modeling, engineering laboratory, and pilot plant studies will be essential to the success of the ultimate plant design.

Competitive-Consecutive Reaction

The first reaction is a bromination that is designed to add one bromine to an aromatic ring but can overreact to add a second bromine. The possibility of reduced selectivity caused by differences in micro- and mesomixing on scale-up must be investigated.

Marco calls one of his associates, Roger, who has looked into this issue in previous developmental studies. Roger advises that the first issue is to decide if the reaction could be influenced by mixing or is reaction-rate controlled. Despite some grumbling that “Mixing cannot affect homogeneous reactions, so why are we doing this?” a geometrically similar 4 liter reactor is set up to study high and low levels of mixing with two feed strategies: addition at the impeller for the high-level case and addition at the surface for the low-level case. No significant difference is observed in the amount of overbromination. Everyone’s first reaction is to jump to the conclusion that there are no mixing issues for this reaction, as was predicted by the grumbling skeptics.

Roger warns that in some cases, a laboratory experiment might not reveal a significant difference because the blend time and micromixing on this scale could be sufficient to mask the problem. He proceeds to recommend further experiments: a reverse addition in which A is added to a solution of the brominating reagent instead of the brominating reagent being added to A, as the chemist’s procedure specifies. The skeptics again object—even more forcefully—because it is obvious that this is no way to run the reaction. Marco asks Roger to explain his reasoning. “This extreme change will almost certainly reveal whether or not there is potential for overbromination since A is added into a sea of bromine reagent. This will exaggerate any overreaction.” Roger notes that the 1 : 1 mole ratio will be maintained once the addition is completed. He also reminds Marco that even a small amount of dibromo (<1%) could be a problem since scale-up will increase the overreaction unless precautions for adequate mixing are recognized and taken. After some consideration, Marco decides that it is easier and cheaper to run the reverse addition experiment than to run the risk of overbromination.

The reverse addition revealed overreaction, indicating to Roger that further testing on a pilot plant scale would be required. After determination of the reaction rate ratio, k_1/k_2 , Roger was concerned that successful scale-up in a stirred vessel might not be feasible because the consecutive overbromination reaction was relatively fast. A high-energy pipeline mixer reactor might be required for manufacturing. The skeptics were still not impressed, figuring that Roger was making a big deal out of a small problem.

For a discussion of mixing issues for this type of reaction, and more on who was right, Roger or the skeptics, the reader is referred to Chapters 13 and 17 on reactor design as well as Chapter 2 on homogeneous turbulence and Chapter 9 on blending in tanks. This particular issue, and a vindication of Roger's position, is detailed in Example 13-1.

Gas-Liquid Reaction

Moving along with his analysis, Marco notes that the second reaction is a hydrogenation. The chemist was running the hydrogenation in a 1 liter autoclave over a period of 8 hours (or sometimes overnight, thereby avoiding several evenings in the laboratory). Marco immediately suspects a mixing limitation on the gas-liquid contact even though the chemist adamantly maintained that it was just a slow reaction.

Mary, his expert in these types of reactions, has seen a similar issue before. She concurs that the mixing conditions in the autoclave will lead to inadequate hydrogen absorption. Surface reincorporation is not being achieved because of the design of the autoclave—high z/T ratio and full baffles—a common but poor laboratory autoclave mixing system. Mary sets up a modified mixing system for the autoclave by cutting the baffles to create a vortex to the top pitched blade turbine in order to achieve surface reincorporation of hydrogen that has not reacted after sparging but has escaped to the headspace. By escaping to the headspace and building up pressure, this hydrogen effectively reduces or stops hydrogen flow into the vessel.

In Mary's modified autoclave, the reaction takes off and is complete in 30 minutes, much to the chemist's surprise. In making such a reactor modification, it is wise to prepare for more rapid heat evolution than was experienced in the improperly mixed original autoclave. Mary was prepared and had no difficulty controlling the temperature. It is also necessary to provide good bearing support for the shaft to counteract the increased vibration associated with vortexing.

The next issue for Marco to consider is how to scale-up this reaction for the pilot plant and production plant. Discussion of this type of reaction and the associated scale-up issues may be found in Chapters 13 and 17. Gas-liquid mixing issues are discussed in Chapter 11.

Solid-Liquid Reaction

The next reaction in the process is an alkylation using powdered potassium carbonate as a base to react with an organic acid reagent (in solution) to form the potassium salt (in solution). This reaction appears to be very straightforward and is transferred directly to the pilot plant. After the first pilot plant run, Marco gets an e-mail: the reaction was slower than expected and resulted in incomplete conversion. In addition to this, the operators spent the rest of the shift getting the batch out of the vessel because the bottom outlet was clogged with solids. Marco immediately suspects the culprit—inadequate off-bottom suspension of

the powdered potassium carbonate. Marco explains that in a dissolving reagent reaction the solid dissolution step can be rate controlling. When the reaction time is extended to compensate for this, simultaneous decomposition of the product (in solution) can be enhanced. Another possibility is decomposition caused by contact of the base with the product in the high-pH liquid film around the particles. Marco recommends that the first issue to address is that all of the powder did not even get to react because it was mounded on the bottom of the vessel. To fix this problem, adequate mixing for off-bottom suspension is essential. In addition, improved mixing will favor rapid dissolution of the highly insoluble K_2CO_3 . Marco cautions that overmixing could cause foaming in this type of fine-solid–liquid suspension. This may cause the particles to be coated with inert gas (nitrogen) and therefore have reduced dissolution characteristics. The particle size of the K_2CO_3 also has a significant effect on the dissolution time, so a change in supplier or grade of solids could be the problem, although in this case it is not.

Marco sets up a 4 liter reaction flask with a pitched blade turbine impeller and full baffles. Experiments are run to determine the effects of K_2CO_3 particle size and impeller speed on reaction rate over wide ranges. The sensitivity of reaction rate to both particle size and impeller speed is readily established. The loss of product due to decomposition is also demonstrated.

These results illustrate the critical nature of solids dissolution of reagents in chemical reactions. The reader is referred to Chapters 13 and 17 for discussion and further examples as well as to Chapter 10 for calculation of solids suspension requirements.

Liquid–Liquid Reaction

Six months later, Marco and his group have solved the three reaction problems outlined above by experimentation and by studying the appropriate literature references, including applicable parts of this handbook. The liquid–liquid reaction issues for the fifth reaction in the synthesis have also been solved in the laboratory and pilot plant and successfully scaled-up to manufacturing. This brings us to the phone call from Bill that we overheard at the beginning of this story. Manufacturing moved the reaction to a different vessel, causing a drop in conversion rate and selectivity in Step 5. As often happens, there are compelling reasons that prevent Bill from solving his problem by going back to the original, successful reactor, just as there were compelling reasons to change reactors in the first place. It may become obvious, however, that the compelling reasons are not as compelling as they may seem when a vessel transfer that appears straightforward turns into a nightmare for the plant.

On the phone, Bill indicates that the mixing still looks good in the new vessel but does acknowledge that the new vessel has a different impeller design and speed. Before leaving, Marco grabs a coffee with Vijay, who did some work in this area in graduate school. They agree to follow the mixing trail despite Bill's visual characterization of the mixing as good. As he sits on the plane to Texas, Marco wonders, "What could be causing slower reaction and increased

by-product formation? A change in the mixing characteristics between the two reactors?” Marco remembers his anxiety during development of this liquid–liquid reaction because he was well aware of the potential for difficulty. He was very pleased that it had been piloted and scaled up to manufacturing successfully—at least until the change of vessel.

Liquid–liquid complex reactions have been classified as one of the most difficult—if not *the* most difficult—reaction scale-up mixing problem (Leng, 1997). The complexities of drop formation and coalescence both change with scale. They both depend on the location in a vessel and on very subtle changes in the composition of the fluids. These variations can cause problems like Bill’s when a complex reaction occurs between reagents in separate liquid phases.

When Marco arrives at the plant, Bill suggests that the most readily achievable “fix” is to increase impeller speed. He can accomplish this with a change in drive gears, although a higher-power motor will be required. Should the increase in rpm be based on equal P/V, on equal tip speed, or on something else? Marco and Vijay know that an increase in rpm with a different impeller might not work since geometric similarity cannot be maintained. Marco was also harboring the disturbing thought that overmixing, by providing too much power while improving reaction rate, could actually reduce selectivity by exposing product in the droplet films to high concentrations of reagent in the aqueous phase. Is this possible? Has it ever been experienced?

One dilemma in answering these questions is that laboratory scale experimentation may not be able to provide a suitable model for scale-up. Bill, Vijay, and Marco may have to make a decision on the fix without quantitative information. Fortunately, most mixing problems can be addressed with more certainty than those involving fast, complex reactions in multiple phases. These issues are discussed in Chapters 13 and 17 as well as in Chapter 12 (liquid–liquid mixing). In addition, comparisons between impellers and general information on the components of stirred vessels may be found in Chapter 6, and the help that can be provided by mixing equipment suppliers is discussed in Chapter 22.

Crystallization

At the outset of this project, Marco noted that the seven-step synthesis includes four crystallization operations. One of these is a final reactive crystallization that will determine the physical attributes of the product. Any of these steps could produce crystals that are difficult to filter, wash, and dry because the particle size distribution could change on scale-up due to mixing effects during crystallization. Excess nucleation and/or crystal fracture are both expected to be more severe in plant operation than in the laboratory or even the pilot plant. From a process design point of view, elimination of one or more of the crystallization steps will yield large savings in both capital and operating costs. However, discussion of this type of development initiative is not included in the scope of this book because mixing issues would not be primary considerations in developing these strategies.

The reactive crystallization that is the last step of this complex process could present critical mixing issues because mixing can affect both the reaction and

subsequent crystallization. The physical attributes and chemical purity of the final product will be determined by the success or failure of the scale-up. To add to this complexity, intense mixing may be required for the fast reaction, whereas modest blending may be simultaneously required to prevent crystal attrition. These mutually exclusive requirements require a compromise to achieve the best result possible.

Initially, Marco was at a bit of a loss because his group had always had difficulty with reactive crystallizations and had not developed a successful strategy for overcoming the basic issues inherent to this type of crystallization (also termed *precipitation*). Since these operations are almost always carried out at high supersaturation, they are nucleation based and therefore tend to produce small crystals, typically 5 to 10 μm in size, with many in the 1 μm range. Both occlusion of impurities and unacceptable physical attributes can result. Marco assigned Carol, a new engineer, to work with Joe, a veteran of many crystallization developments, who remained hopeful that this dilemma of precipitation could be solved.

Carol and Joe succeeded in balancing the reaction requirements with the crystallization parameters required to achieve a growth-dominated process. In doing so, they had to choose a mixing system that would achieve micromixing effectively for the fast reaction but which was compatible with crystal growth. Mixing issues in this and other types of crystallization operations are discussed in more detail in Chapter 17.

USING THE HANDBOOK

This book is not meant to be read from beginning to end. It is designed as a reference, with extensive cross-referencing and indexing. The book is divided into three sections: fundamentals, design, and applications. Many examples are included to aid the reader in understanding the fundamentals as well as some case histories of mixing issues in industrial practice. Authorship of most of the chapters includes both academic and industrial contributors, for the purpose of providing a broad perspective on each topic. Also included is a CD ROM to aid in visualization of some specific mixing issues and examples. The sections in this introduction should help the reader new to the field of mixing in identifying what is meant by a mixing problem. We have summarized key issues (Mixing in Perspective) and discussed a process containing examples of many reactive mixing problems (Conversations Overheard in a Chemical Plant), and diagnostic charts follow this summary. All of these sections provide the reader with references to relevant chapters in the handbook.

The text and examples include guidance in troubleshooting mixing problems based on understanding the fundamental issues, aided by drawing on the experiences cited. It is often assumed that mixing scale-up is accomplished by direct scaling to a larger pot. This approach may work in some cases but is doomed to failure in others. The key question is the determination of process requirements for which direct scale-up will be inadequate. Another overall concern is

to beware of the fact that multiple process objectives often must be realized in a single piece of mixing equipment, thereby requiring selection of a design basis compatible with the most critical scale-up issue(s).

Diagnostic Charts

Figures I-1 through I-6 summarize some of the key symptoms and causes of mixing problems in the plant. They are in no way exhaustive and should not be used to replace the expertise of an experienced process specialist. They can be used to guide you through some of the implicit steps used in evaluating mixing problems, and may help to focus your reading.

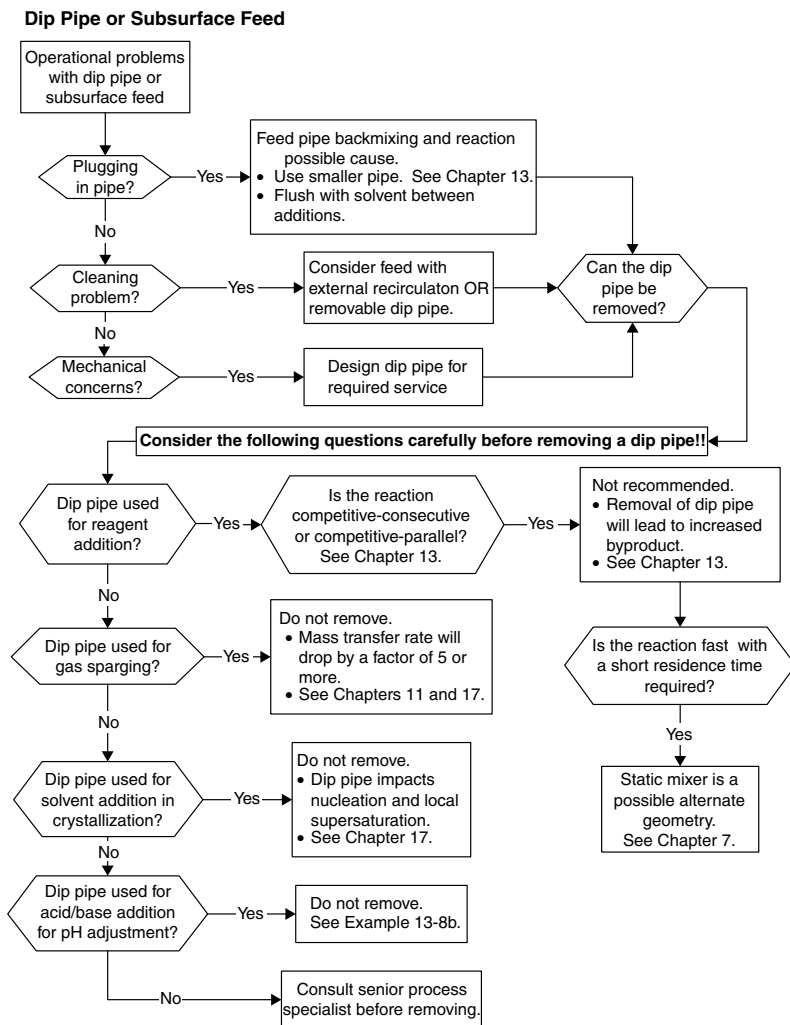


Figure I-1 Dip pipe or subsurface feed.

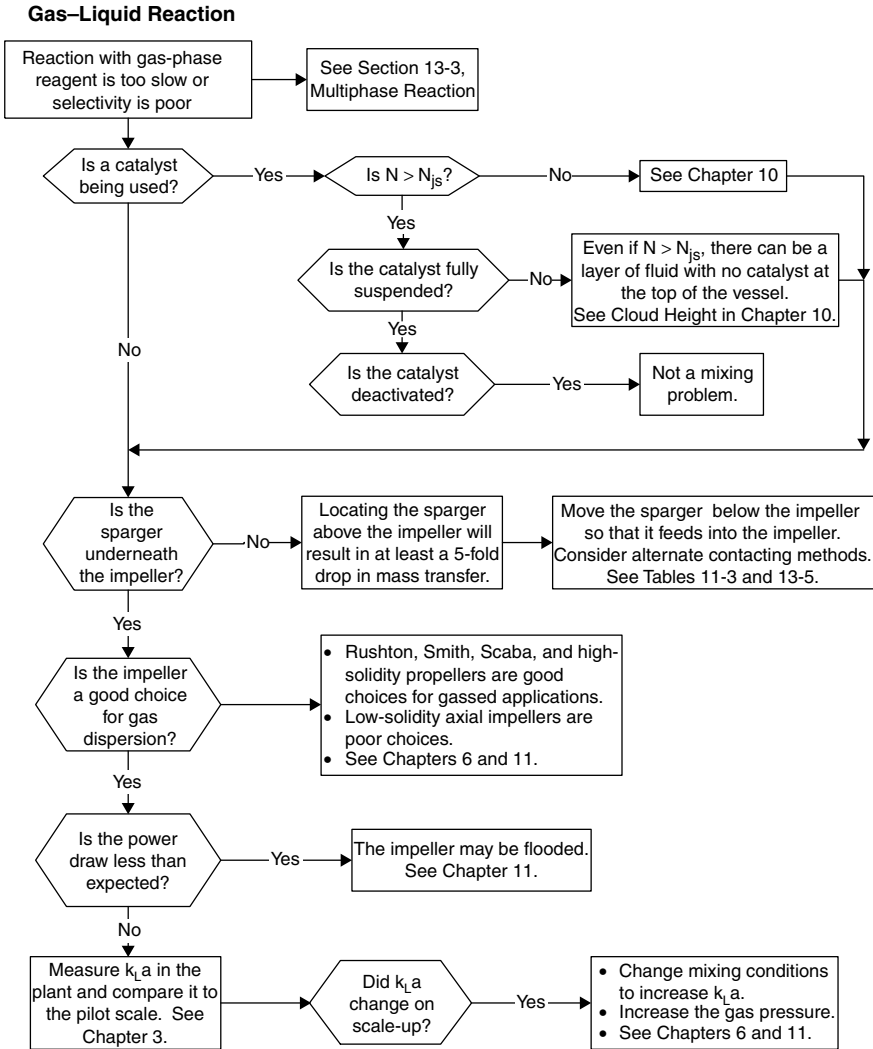


Figure I-2 Gas-liquid reaction.

Batch Liquid-Liquid Extraction

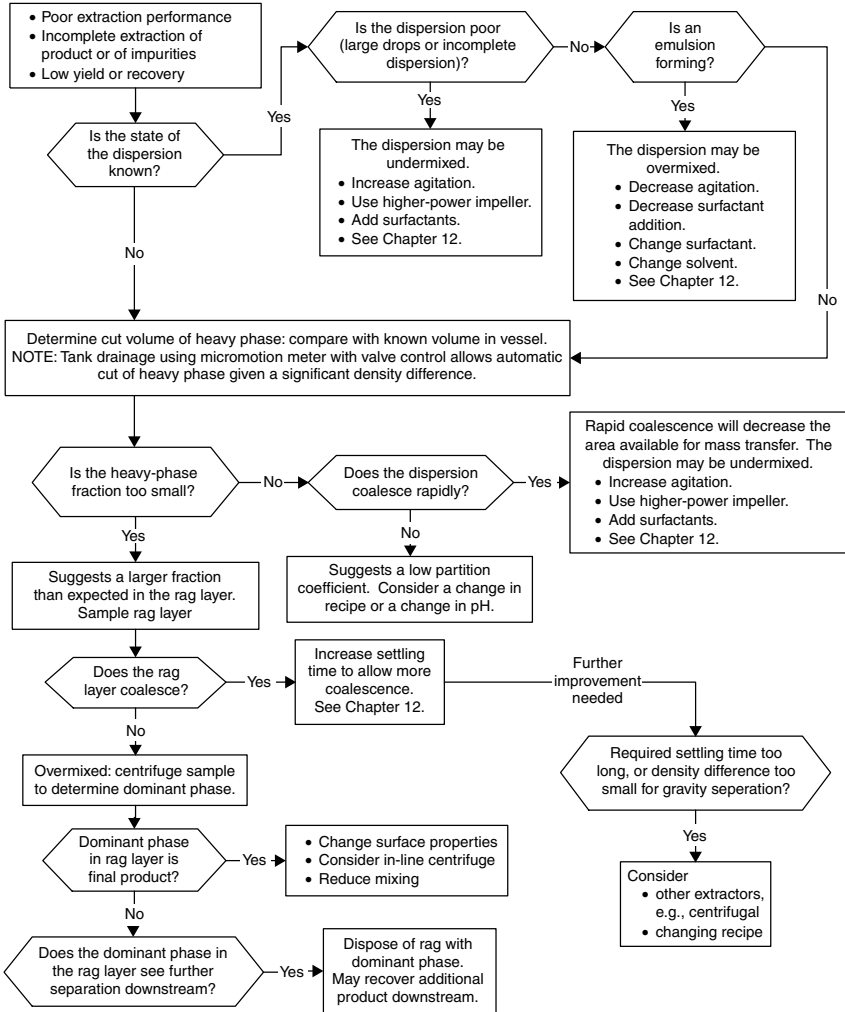


Figure I-3 Batch liquid-liquid extraction.

Reaction in Liquid-Liquid Dispersion

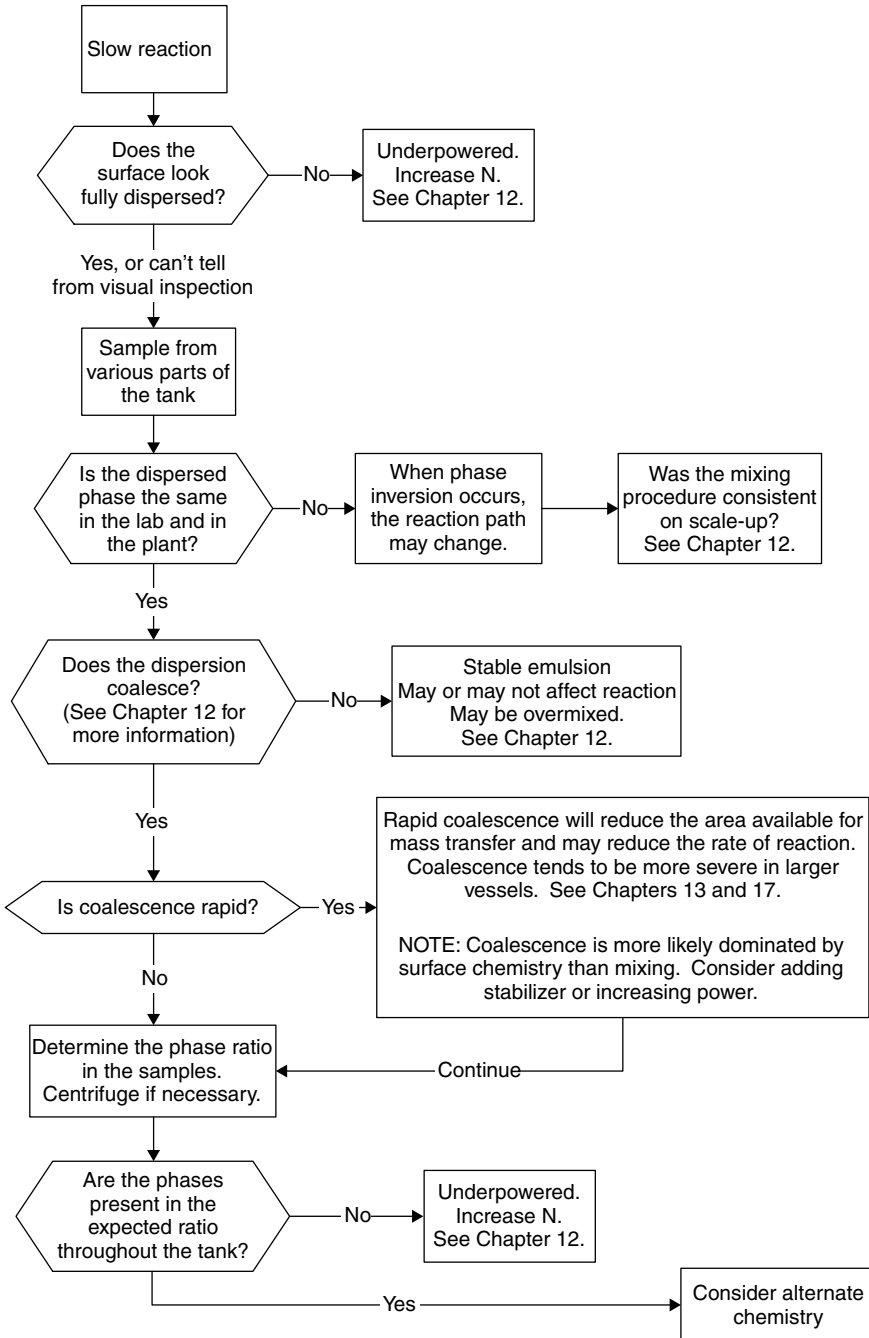
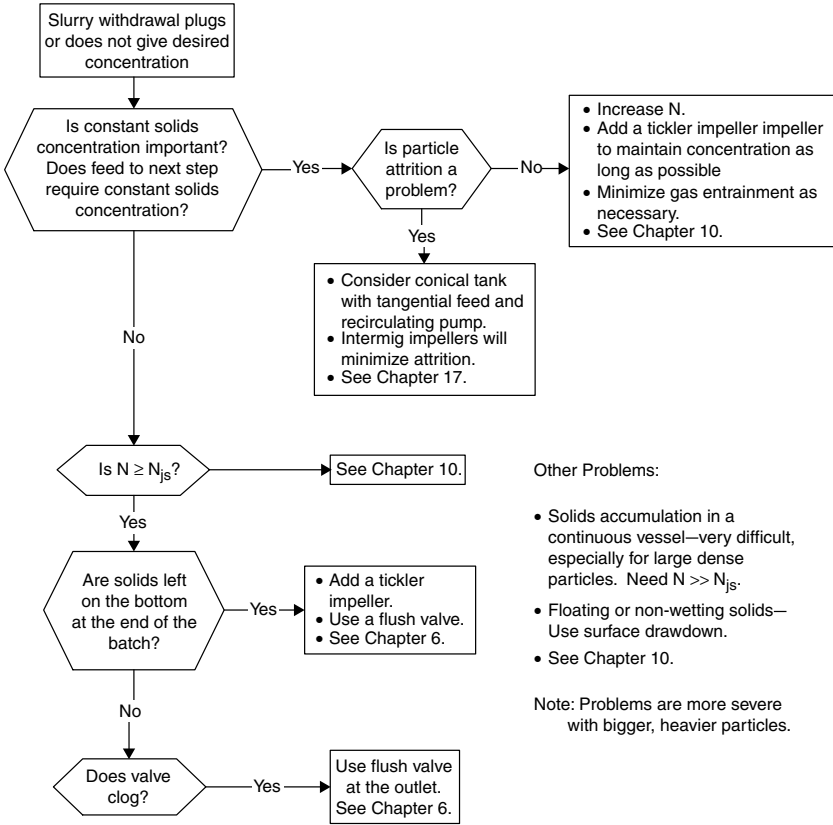


Figure I-4 Reaction in liquid-liquid dispersion.

Solids Withdrawal from Stirred Tanks



Other Problems:

- Solids accumulation in a continuous vessel—very difficult, especially for large dense particles. Need $N \gg N_{js}$.
- Floating or non-wetting solids—Use surface drawdown.
- See Chapter 10.

Note: Problems are more severe with bigger, heavier particles.

Figure I-5 Solids withdrawal from stirred tanks.

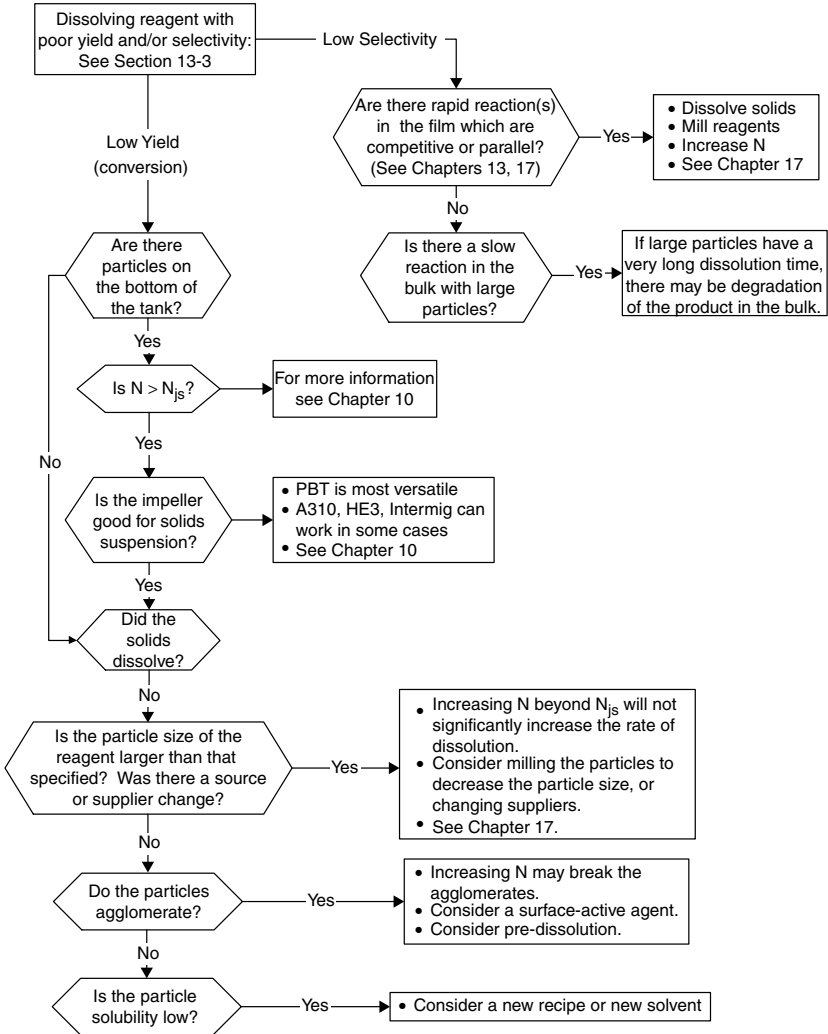
Solid-Liquid Reaction**Figure I-6** Solid-liquid reaction.**Mixing Nomenclature and Unit Conversions**

Table I-1 includes the common nomenclature used in mixing correlations and calculations. Many of the chapters in this book have more detailed lists of nomenclature for specific topics. Table I-1 is intended for general reference. Where a symbol is used for more than one purpose, the common multiple uses are given. Conversions are given in Tables I-2 and I-3.

The nomenclature follows that outlined by Oldshue (1977), Buck (1978), and the AIChE Equipment Testing Procedure for Mixing Equipment (2001) of symbols for use in the SI system. There are a few exceptions that are commonly

Table I-1 Mixing Nomenclature

| Common Symbol | Quantity | Units |
|-------------------|--|---|
| A, B, R, S | Reactants | |
| A, B, R, S | Reactant concentrations, C_A , C_B , etc. | mol/m ³ |
| B | Baffle width | m |
| C | Impeller off-bottom clearance | m |
| C | Reaction conversion, $(A_o - A)/A_o$ | % |
| C_p | Specific heat | J/kg · K |
| D | Impeller diameter | m |
| D_{AB} | Diffusivity | m ² /s |
| Da | Damkohler number (see Chapter 13) | (-) |
| d_{32} | Sauter mean diameter | m |
| Fr | Froude number, N^2D/g | (-) |
| g_c | Gravitational correction for British units, $32.2 \text{ lb}_m/\text{lb}_f \times \text{ft}/\text{s}^2$ | |
| H or Z | Liquid height | m |
| k_1, k_2, \dots | Reaction rate constants | (mol/m ³) ¹⁻ⁿ /s |
| k | Thermal conductivity | W/m · K |
| k_g, k_l | Mass transfer coefficient | m/s |
| L | Length scale | m |
| N | Impeller rotational speed | rps or rpm |
| N_c | Impeller critical rotational speed | rps or rpm |
| N_{js} | Just suspended rotational speed | rps or rpm |
| N_{min} | Just suspended speed for liquid drops | rps or rpm |
| Nu | Nusselt number, hT/k | (-) |
| N_p or Po | Power number, $P_{gc}/\rho N^3 D^5$ | (-) |
| P | Power, $N_p \rho N^3 D^5 / g_c$ | W |
| P | Pressure | Pa |
| Pr | Prandtl number, $C_p \mu / k$ | (-) |
| Q | Heat transfer rate, $UA\Delta T_m$ | W |
| Q_L | Pumping rate of impeller, αND^3 | m ³ /s |
| R | Gas constant | J/mol · K |
| R | Impeller radius | m |
| Re | Reynolds number, $\rho DV/\mu$ | (-) |
| Re | Impeller Reynolds number, $\rho D^2 N/\mu$ | (-) |
| S | Reaction selectivity (see Chapter 13) | |
| Sc | Schmidt number, $\mu/D_{AB}\rho$ | (-) |
| T | Tank diameter | m |

Table I-1 (continued)

| Common Symbol | Quantity | Units |
|-------------------------------|--|--------------------------------|
| T | Temperature | K,C |
| t | Time | s |
| To or T _Q | Torque, $\alpha\rho N^2 D^5$ | W/s |
| U | Overall heat transfer coeff, $Q/(A \Delta T_m)$ | $J/m^2 \cdot s \cdot K$ |
| u' | Fluctuating velocity | m/s |
| V _{impeller} | Impeller swept volume | m ³ |
| V | Volume | m ³ |
| V | Velocity | m/s |
| W _b | Baffle width | m |
| X _s | Impurity selectivity, 2S/R + 2S | % |
| Y | Reaction yield, R/A _o | % |
| Z and H | Liquid height | m |
| <i>Greek Symbols</i> | | |
| α | Blade angle | ° |
| γ | Shear rate | s ⁻¹ |
| δ | Width of shear gap, rotor and stator | m |
| ε | Void fraction | (-) |
| ε | Local rate of dissipation of turbulent kinetic energy per unit mass | m ² /s ³ |
| $\bar{\varepsilon}$ | Power input per mass of fluid in the tank, power per volume, $P/\rho V_{\text{tank}}$ | m ² /s ³ |
| $\bar{\varepsilon}_i$ | Power input per mass of fluid in the impeller swept volume, $P/\rho V_{\text{impeller}}$ | m ² /s ³ |
| η (also λ_K) | Kolmogorov scale, $(\nu^3/\varepsilon)^{1/4}$ | m |
| $\theta_B, t_{\text{blend}}$ | Blend time | s |
| θ | Angle of impeller blade with axis of rotation | ° |
| λ | Taylor microscale of turbulence | m |
| λ | Wavelength | m |
| λ_B | Bachelor length scale, $(\nu D_{AB}^2/\varepsilon)^{1/4}$ | m |
| λ_K (also η) | Kolmogorov scale, $(\nu^3/\varepsilon)^{1/4}$ | m |
| μ | Viscosity | Pa · s |
| ν | Kinematic viscosity, μ/ρ | m ² /s |
| ρ | Density | kg/m ³ |
| σ | Interfacial tension | N/m |
| τ_M | Mixing time constant | s |
| τ_D | Diffusion time constant | s |
| τ_R | Reaction time constant | s |
| τ | Shear stress | Pa |
| τ (also T _Q) | Torque | N · m |
| ϕ | Volume fraction of dispersed phase | (-) |
| ϕ | Particle shape factor | (-) |

Table I-2 Conversion from British to SI Units

| Non-SI Unit | Quantity | To Convert to SI Unit: | Multiply by: |
|-------------------------------------|-----------------------------|------------------------------|----------------|
| Btu | Heat | Joule (J) | 1.0551 E +03 |
| Btu/lb _m · °F | Heat capacity | J/kg · K | 4.1868 E +3 |
| Btu/hr | Heat flux | Watt (W) | 2.9307 E -01 |
| Btu/hr · ft ² · °F | Heat transfer coefficient | W/m ² · K | 5.6782 E +00 |
| Btu/ft · hr · °F | Thermal conductivity | W/m · K | 1.7307 E +00 |
| cal | Calorie | Joule (J) | 4.1868 E +00 |
| centipoise | Viscosity | Pa · s | 1.0000 E -03 |
| centistoke | Kinematic viscosity | m ² /s | 1.0000 E -06 |
| °F | Temperature | C | (°F - 32)(5/9) |
| dyne | | Newton (N) | 1.0000 E -05 |
| erg | | Joule (J) | 1.0000 E -07 |
| foot | | meter (m) | 3.0480 E -01 |
| foot ² | | m ² | 9.2990 E -02 |
| foot ³ | | m ³ | 2.8316 E -02 |
| ft · lb _f | | Joule (J) | 1.3558 E +00 |
| gallon | U.S. liquid | m ³ | 3.7854 E -03 |
| horsepower | 550 ft·lb _f /sec | Watt (W) | 7.4570 E +02 |
| inch | | meter (m) | 2.5400 E -02 |
| inches Hg (60°F) | | Pascal (Pa) | 3.3768 E +03 |
| inches H ₂ O (60°F) | | Pascal (Pa) | 2.4884 E +02 |
| kilocalorie | | Joule (J) | 4.1868 E +03 |
| micron | | meter (m) | 1.0000 E -06 |
| mmHg (0°C) | Pressure | Pascal (Pa) | 1.3332 E +02 |
| poise | Absolute viscosity | Pa · s | 1.0000 E -01 |
| lb _f | | Newton (N) | 4.4482 E -00 |
| lb _m | | kilogram (kg) | 4.5359 E -01 |
| lb _m /ft ³ | Density | kg/m ³ | 1.6018 E +01 |
| lb _m /ft-sec | Viscosity | Pa · s | 1.4882 E +00 |
| psi | Pressure | Pascal (Pa) | 6.8948 E +03 |
| Stoke | Kinematic viscosity | m ² /s | 1.0000 E -04 |
| tonne (long, 2240 lb _m) | | kilogram (kg) | 1.0160 E +03 |
| ton (short, 2000 lb _m) | | kilogram (kg) | 9.0718 E +02 |
| torr (mmHg, 0°C) | Pressure | Pascal (Pa) | 1.3332 E +02 |
| Watt | | Watt (W) | 1.0002 E +00 |
| Watt-h | | Joule (J) | 3.6000 E +03 |

Table I-3 Conversion of SI Units

| SI Unit | To Convert to | Multiply by: |
|--------------------------|-------------------------|--------------|
| Joule (J) | Btu | 9 E -4 |
| | ft-lb _f /sec | 0.7375 |
| Watt (W) | Btu/hr | 3.436 |
| Volume (m ³) | ft ³ | 35.32 |
| | liter | 1000 |
| | gallon | 264.2 |
| Meter (m) | angstrom | 1.000 E +10 |
| | micron (μm) | 1.000 E +6 |
| Viscosity (Pa · s) | centipoises | 1.000 E +3 |
| Power (W) | horsepower | 0.0013 |
| Pressure (Pa) | inch Hg | 0.2953 E -3 |
| | psi | 0.1451 E -3 |
| | torr (mmHg at 0 K) | 7.5006 E -3 |

used in mixing terminology. The European Federation of Chemical Engineering Working Party on Mixing Terms, Symbols, and Units has also published a comprehensive list of nomenclature (Fort et al., 2000).

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The objective of this book is to provide mixing practitioners with a summary of the current state of mixing knowledge, both in terms of fundamentals and from the perspective of industrial practice and experience. We have found many of the chapters absolutely definitive in their area. We hope that readers find as much to stimulate and fascinate them in these pages as we have found during their editing.

As we complete the final pages of this manuscript, we would like to thank our families for their unwavering faith and encouragement during its preparation. At times the book appeared to be a fantasy of our own creation, and at times the work has been very real and seemingly unending. Through it all, they have shared our road less traveled by; and that made all the difference.

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