INTRODUCTION

It should be noted that the coverage of this section and that of the next one (8.10) are similar. If you are interested in reading a detailed and complete treatment of the subject of chemical reactor control, read this section. On the other hand, if you have little time and are an experienced process control engineer, familiar with the basics of chemical reactor control, and want only to refresh your memory about the most important aspects of their control and optimization, read Section 8.10.

This section is started with a description of reactor characteristics, reaction rates, and time constants. This is followed by a discussion of the various methods of reactor temperature control, initial heat-up control, end-point detection, pressure, and safety controls. Other aspects of reactor control are covered in Sections 8.8, 8.10, and 8.11, covering the topics of batch sequencing, optimization, and modeling of chemical reactor controls.

Chemical reactor designs include the continuous stirred tank reactors (CSTRs), the batch stirred tank reactors (BSTRs), the tubular reactors, and the packed bed reactors. The optimization of batch and continuous chemical reactors has many potential benefits, including increase in productivity and improvement in safety, product quality, and batch-to-batch uniformity. The combined impact of these factors on plant productivity can approach a 25% improvement.¹

Such overall results are the consequences of many individual control loops and control strategies. These loops will program temperature and pressure and maintain concentration and safety, while providing sequencing and record-keeping functions. All elements of the overall chemical reactor control system are discussed in this chapter.

REACTOR DESIGNS AND CHARACTERISTICS

In a batch cycle, there is no steady state and therefore no “normal” condition at which controllers could be tuned. The dynamics of the batch process vary with time; thus, the process variables, the process gains, and time constants also vary during the batch cycle. In addition, there are the problems of runaway reactions and batch-to-batch product uniformity.

Runaway reactions occur in exothermic reactions, in which an increase in temperature speeds up the reaction, which in turn releases more heat and raises the temperature further. In order to counter this positive feedback cycle, highly self-regulating cooling systems are required. One of the most self-regulating cooling systems is a bath of boiling water, because it needs no rise in temperature to increase its rate of heat transfer. Endothermic reactions are inherently self-regulating.

Batch-to-batch uniformity is a function of many factors, from the purity of reactants, catalysts, and additives to the repeatability of controllers serving to maintain heat and material balance. Before addressing such complex topics, it is necessary to review the basic batch process.

Most batch cycles are started by charging reactants into the reactor and then mixing and heating them until the reaction temperature is reached. The reaction itself is frequently started by the addition of a catalyst. Exothermic reactions produce heat, and endothermic reactions consume heat. The reactor itself can be isothermal, meaning that it is operated at constant temperature, or adiabatic, meaning that heat is neither added nor removed within the reactor; the reaction is controlled by other means, such as the manipulation of pressure, catalyst, and reactants.

Chemical reactions can follow quite complex paths and sequences, but for engineering purposes such as equipment design and control system analysis, most reactions can be considered as one of four types: irreversible, reversible, consecutive, or simultaneous. Most reactions are reversible — that is, there is a ratio in product-to-reactant concentration that brings about equilibrium. Under equilibrium conditions the production rate is zero, because for each molecule of product formed there is one that converts back to its reactant molecules.

The equilibrium constant (K) describes this state as the ratio of forward- to reverse-rate coefficients. The value of K is also a function of the reaction temperature and the type of catalyst used. K naturally places a limit on the conversion...
that can be achieved within a particular reactor, but conversion can usually be increased, if at least one of the following changes can be made:

- Reactant concentration can be increased
- Product concentration can be decreased through separation or withdrawal
- Temperature can be lowered by increased heat removal in reversible exothermic reactions
- A change in operating pressure can be affected (this increases conversion only in certain reactions)

The catalyst does not take part in the reaction, but it does affect the reaction rate ($k$). Some catalysts are solids and are packed in a bed; others are fluidized, dissolved, or suspended. Metal catalysts are frequently formed as flow-through screens. Whatever their shape, the effectiveness of the catalyst is a function of its active surface, because all reactions take place on that surface. When it is fouled, the catalyst must be reactivated or replaced.

The time profiles of heat release, operating temperature, and chemical concentrations are illustrated in Figure 8.9a for a consecutive reaction, in which first ingredient $A$ is converted into intermediate product $B$, and then intermediate product $B$ reacts to form final product $C$. The reaction temperature is controlled so as to maximize the production of $C$ while minimizing the cycle period.

**Reaction Rates and Kinetics**

The reaction rate coefficient exponentially increases with temperature. The activation energy ($E$) determines its degree of temperature dependence according to the Arrhenius equation:

$$k = \alpha e^{-(E/R)T}$$  \hspace{1cm} 8.9(1)

where

- $k$ = specific reaction rate coefficient (min$^{-1}$)
- $\alpha$ = pre-exponential factor (min$^{-1}$)
- $E$ = activation energy of reaction (BTU/mole)
- $R$ = perfect gas constant (1.99 BTU/mole·°R)
- $T$ = absolute temperature (°R)

Figure 8.9b illustrates the strong dependency of the reaction rate coefficient ($k$) on reaction temperature for the values of $\alpha = e^{29}$ and $E/R = 20,000$.

Figure 8.9c illustrates the three basic reactor types: 1) plug flow, 2) continuous stirred tank, and 3) batch; it mathematically defines their fractional conversion of the reactant(s) into product ($y$).

Because the continuous plug flow-type reactor is dominated by dead time, its temperature control is difficult. On the other hand (as shown in Figure 8.9c), the plug flow reactor gives higher conversion than a back-mixed reactor operating under the same conditions. If the reaction rate is low, a long tubular reactor or a larger back-mixed reactor is required to achieve reasonable conversions.

In a batch reactor, after the initial charge there is no inflow or outflow. Therefore, an isothermal batch reactor is similar in its conversion characteristics to a plug-flow tubular reactor. If the residence times are similar, both reactors will
provide the same conversions. Batch reactors are usually selected when the reaction rates are low, when there are many steps in the process, when isolation is required for reasons of sterility or safety, when the materials involved are hard to handle, and when production rates are not high.

As shown in Figure 8.9d, the batch (or tubular) reactor is kinetically superior to the continuous stirred tank reactor. The batch reactor has a smaller reaction time and can produce the same amount of product faster than the back-mixed one.

**Reactor Time Constants**

The amount of heat generated by an exothermic reactor increases as the reaction temperature rises. If the reactor is operated without a temperature controller (in an open loop), an increase in the reaction temperature will also increase heat removal, because of the increase in $\Delta T$ between process and coolant temperatures. If an increase in reaction temperature results in a greater increase in heat generation than in heat removal, the process is said to display positive feedback; as such, it is considered to be “unstable in the open loop.”

The positive feedback of the open-loop process can be compensated for by the negative feedback of a reactor temperature controller, which will increase the heat removal rate as the temperature rises. The addition of such a feedback controller can stabilize an open-loop unstable process only if the control loop is fast and does not contain too much dead time. Cascade control can increase speed, and maximized coolant flow can reduce dead time. Shinskey suggests that if the dead time can be kept under 35% of the thermal time constant of the reactor, the process can be stabilized, whereas if it approaches 100%, the reactor will not be controllable.

A real reactor has several lags and delays, including those of measurement and heat removal, as illustrated in Figure 8.9e.
The equations for calculating the thermal, reactor wall, coolant, and thermal bulb time delays are listed below. Typical values of these time constants are:

\[
\begin{align*}
\tau_1 &= \text{thermal time constant} = 30-60 \text{ min} \\
\tau_2 &= \text{reactor wall time constant} = 0.5-1.0 \text{ min} \\
\tau_3 &= \text{coolant time constant} = 2-5.0 \text{ min} \\
\tau_4 &= \text{thermal bulb time constant} = 0.1-0.5 \text{ min (can be minimized by the use of bare bulbs)}
\end{align*}
\]

Thermal time constant: \[
\tau_1 = \frac{W_1 C_1}{k_1 A} = \frac{W_1 C_1}{Q} (T - T_1) \tag{8.9(2)}
\]

Reactor wall time constant: \[
\tau_2 = \frac{W_2 C_2}{k_2 A} = \frac{W_2 C_2}{Q} (T_2 - T_2) \tag{8.9(3)}
\]

Coolant time constant: \[
\tau_3 = \frac{W_3 C_3}{k_3 A} = \frac{W_3 C_3}{Q} (T_3 - T_3) \tag{8.9(4)}
\]

Thermal bulb time constant: \[
\tau_4 = \frac{W_4 C_4}{k_4 A} \tag{8.9(5)}
\]

where

- \(A\) = heat-transfer area, ft\(^2\)
- \(A_s\) = surface area of bulb, ft\(^2\)
- \(C\) = specific heat of coolant, BTU/(lb)(°F)
- \(C_1\) = specific heat of reactants, BTU/(lb)(°F)
- \(C_2\) = specific heat of wall, BTU/(lb)(°F)
- \(C_3\) = specific heat of bulb, BTU/(lb)(°F)
- \(k_1\) = heat-transfer coefficient, BTU/(h)(ft\(^2\))(°F)
- \(k_2\) = thermal conductivity, BTU/(h)(ft)(°F/in)
- \(k_3\) = heat-transfer coefficient, BTU/(h)(ft\(^2\))(°F)
- \(l\) = wall thickness, in.
- \(Q\) = rate of heat evolution, BTU/h
- \(T\) = reactor temperature, °F
- \(T_1\) = wall temperature, °F
- \(T_2\) = outside wall temperature, °F
- \(T_3\) = average coolant temperature, °F
- \(W_1\) = weight of reactants, lb
- \(W_2\) = weight of wall, lb
- \(W_3\) = weight of jacket contents, lb
- \(W_4\) = weight of bulb, lb

Temperature is not uniform and the process dead time varies with load. In the case of a typical reactor, the period of oscillation might be around 30 min. This period approximately equals four dead times; therefore, the total dead time of such a loop is around 7.5 min. In the case of interacting controllers, the correct setting for such a controller would be 7.5 min for both integral and derivative times.

Two types of reactors are used in chemical plants: continuous reactors and batch reactors. Continuous reactors are designed to operate with constant feed rate, withdrawal of product, and removal or supply of heat. If properly controlled, the composition and temperature can be constant with respect to time and space. In batch reactors, measured quantities of reactants are charged in discrete quantities and allowed to react for a given time, under predetermined controlled conditions. In this case, composition is the function of time.

**TEMPERATURE CONTROL**

The control loop features required during heat-up are substantially different from those needed during an exothermic reaction or those required during stripping or refluxing. Each will be discussed in the following paragraphs, starting with the controls of exothermic reactors.

Reaction temperature is frequently selected as the controlled variable in reactor control. It may be necessary to control reaction rate, side reactions, distribution of side products, or polymer molecular weight and molecular weight distribution. All of these are sensitive to temperature. It is frequently necessary to control reaction temperature to within 0.5°F (0.28°C). Many reactions are exothermic. In order to control reaction temperature, the released heat must be removed from the system as it is liberated by the reactants.

A simple temperature control scheme is depicted in figure 8.9f. The reaction temperature is sensed, and the flow of heat-transfer medium to the reactor jacket is manipulated. For many installations this scheme is considered to be unsatisfactory because of the reactor nonlinearity and dynamic features. This “once-through” method of cooling is undesirable.
because the coolant temperature is not uniform. This can cause cold spots near the inlet and hot spots near the outlet.

Another disadvantage of this configuration is the variable residence time of the cooling water within the jacket as the flow rate changes. This causes the dead time of the jacket to vary, which in turn necessitates the modification of the control loop tuning constants as the load varies. In addition, when the water flow is low, the Reynolds number will drop off, and with it, the heat-transfer efficiency will also diminish. Low water velocity can also result in fouling of the heat-transfer surfaces.

For all the above reasons, the recirculated cooling water configuration shown in Figure 8.9g is more desirable, because it guarantees a constant and high rate of water circulation. This keeps the jacket dead time constant, the heat-transfer coefficient high, and the jacket temperature uniform, thereby eliminating cold and hot spots.

The fluid velocity in the reactor jacket is maintained high enough to produce satisfactory film coefficients for heat transfer. The fluid velocity can be further increased by additional jets. In addition, the liquid is circulated at a high enough rate to keep the temperature gradient in the heat-transfer medium, as it passes through the jacket, at a high enough level to maintain the jacket wall temperatures throughout the reactor. This keeps the jacket dead time constant and eliminates fouling of the heat-transfer surfaces.

Because the jacket provides a constant heat-transfer area, when the cooling load is low, the process is sensitive and the process gain is large. As shown in Figure 8.9h, as the load rises, the process gain drops in a nonlinear manner. The variable process gain can be partially compensated for by using a variable gain control valve (equal-percentage valve); thereby, when the process gain drops, the valve gain rises and the total loop gain remains relatively constant.

Figure 8.9i illustrates the temperature response of any uncontrolled chemical reactor to a step change in load, assuming that the coolant is applied in a once-through manner (Figure 8.9f), without recirculation. The solid line depicts the temperature response at low loads, and the dotted line depicts temperature response at high loads.

Both dead time and the process gain increase as the load drops. In other words, at low loads it takes longer for the process to start responding, but once it has, the full response develops quickly. At high loads the opposite is the case.

**Cascade Control**

A superior method of reactor temperature control, a cascade loop, is depicted in Figure 8.9j. Here the controlled process...
variable (reactor batch temperature, hereafter called reactor temperature), whose response is slow to changes in the heat-transfer medium flow (manipulated variable), is allowed to adjust the set point of a secondary loop, whose response to coolant flow changes is rapid. In this case, the reactor temperature controller varies the set point of the jacket temperature control loop.

The purpose of the slave loop is to correct for all outside disturbances, without allowing them to affect the reaction temperature. For example, if the control valve is sticking or if the temperature or pressure of the heat-transfer media changes, this would eventually upset the reaction temperature, if the control system was configured as in Figures 8.9f or 8.9g but not in Figure 8.9j.

This is because in Figure 8.9j the slave would notice the resulting upset at the jacket outlet and would correct for it before it had a chance to upset the master. As pointed out in the detailed discussion of cascade systems in Section 2.6, the process lags should be distributed between master and slave loops in such a way that the time constant of the slave is one tenth that of the master. Cascade loops will not function properly if the master is faster than the slave.

It is preferred that the slave controller be used to maintain the jacket outlet (and not inlet) temperature, because this way the jacket and its dynamic response is included in the slave loop. Another advantage of this configuration is that it removes the principal nonlinearity of the system from the master loop, because reaction temperature is linear with jacket-outlet temperature.

The nonlinear relationship between jacket-outlet temperature and heat-transfer-medium flow is now within the slave loop, where it can be compensated for by an equal-percentage valve, whose gain increases as the process gain drops off (Figure 8.9h). In most instances the slave will operate properly with proportional-plus-derivative or proportional only control, which is set for a proportional band of 10–20%.

The period of oscillation of the master loop is usually cut in half as direct control is replaced by cascade. This might mean a reduction from 40 to 20 min in the period and a corresponding reduction of perhaps 30 to 15% in the proportional band. The derivative and integral settings of an interacting controller would also be reduced from about 10 min to about 5 min. This represents a fourfold overall loop performance improvement.

Using jacket-inlet temperature as an override or measurement for the slave may be useful in cases in which the jacket temperature must be limited, e.g., when the reactor is used as a crystallization unit, or in safety systems that serve to protect glass-lined reactors from thermal shock. Such protection might be needed when hot water is generated by direct injection of steam (see Figure 8.9k).

If it is desired to reduce the heat-up time by applying direct steam heating to the reactor jacket and to use both water and methanol as cooling media in the same system, the configuration in Figure 8.9k can be considered. In order to guarantee that water and methanol will not intermix, even accidentally, in addition to the coolant control valve (TCV-2), tight shut-off on/off valves are provided on both coolants (V3 and V4). Such a control system can be operated in a variety of modes.

In Figure 8.9k five operating modes are listed. These can be implemented with positive interlocks. The return flow path is selected to match the type of coolant supply and is provided with a back-pressure regulator to prevent draining of the jacket.

If heat needs to be added in some phases of the reaction while in other phases it must be removed, the controls must be configured in a two-directional manner. Figure 8.9l depicts a cascade temperature control system with provisions for batch heat-up. The heating- and cooling-medium control valves are split-range controlled, such that the heating-medium control valve operates between 50 and 100% control output signal and the cooling-medium control valve operates between 0 and 50%.

It is important to match the characteristics of the valves (zero point) and to avoid nonlinearity at the transition, which can result in cycling. It is equally undesirable to keep both valves open simultaneously, because it results in energy waste. The control system shown in Figure 8.9l is a fail-safe arrangement, because in case of air failure the heating valve is closed and the coolant valve is opened.

Figure 8.9l also shows an arrangement whereby an upper temperature limit is set on the recirculating heat-transfer-medium stream. This is an important consideration if the product is temperature-sensitive or if the reaction is adversely affected by high reactor wall temperature. In this particular case, the set point to the slave controller is prevented from exceeding a present high-temperature limit.

Another feature shown is a back-pressure control loop in the heat-transfer-medium return line. This may be needed to impose an artificial back-pressure, so that during the heat-up
Control and Optimization of Unit Operations

Limitations of Cascade Control

A positioner is a position controller that detects the opening of the valve and corrects for any deviations between measurement (the mechanically detected position) and set point. In such a hierarchical arrangement, only the master can control its variable independently; the slave controller set points must be freely adjustable to satisfy the requirements of the master.

Reset Windup

Whenever the master is prevented from modifying the set point of the slave—because of a limiter, such as in Figure 8.9m, or because the slave has been manually switched from remote to local set point—reset windup can occur. Reset windup is the integration of an error that the controller is prevented from eliminating. Consequently, the cycle no water leaves the recirculation loop and therefore the pump does not experience cavitation problems.
controller output is saturated at an extreme value. Once saturated, the controller is ineffective when control is returned until an equal and opposite area of error unsaturates it.

This problem is eliminated by the external reset (ER) shown in Figure 8.9m. The external reset signal converts the contribution of the integral mode to just a bias (Equation 2.28[7]) and thereby stops the integral action whenever the slave is not on set point. This feature eliminates the need for switching the master to manual and thereby also eliminates the need for the auto/manual station. In addition, it eliminates reset windup upset due to start-ups, shutdowns, or emergency overrides. Whenever external reset is used the slave must have some integral to eliminate the offset; otherwise, the slave offset would cause an offset in the master.

Another limitation is that the cascade loop will be stable only if each slave is faster than its master. Otherwise, the slave cannot respond in time to the variations in the master output signal, and a cascade configuration will in fact degrade the overall quality of control. A rule of thumb is that the period of oscillation of the slave should not exceed 30% of the period of oscillation of the master loop. This requirement is not always satisfied.

For example, in Figure 8.9m it is important to select valve positioners that are faster than the slave temperature controller on the jacket. Similarly, the jacket control loop should contain less dead time than its master, which would usually not be possible if a once-through piping configuration (Figure 8.9f) is used.

One possible method of reducing the dead time of the cascade slave loop is to move the measurement from the jacket outlet (Figure 8.9) to the jacket inlet. This usually is not recommended, because when this is done, the slave will do much less work because the nonlinear dynamics of the jacket (Figure 8.9h) have been transferred into the master loop.

**Multiple Heat-Transfer Media** The use of a single coolant and single heating media (shown in Figure 8.9m) is often insufficient or uneconomical. If one type of coolant (or heating media) is less expensive than another—for example, the cold water used in the system in Figure 8.9n might be less expensive than the chilled water—it is desirable to fully utilize the first before starting to use the second.

For best performance, the fact that there are three valves should be transparent to the temperature controllers. Their gain should be the same, and their combined range should appear as the straight line in Figure 8.9h. This is not easy to achieve, particularly when the valves are nearly closed (see Figure 6.7e in Chapter 6 for a discussion of valve gains), which happens to be the case when the controller output is 11 PSIG in Figure 8.9n.

Therefore, some users prefer to provide some overlap so that the water valve might start opening at 11 PSIG, while the steam valve does not close fully until the signal drops to 10.5 PSIG. Overlapping at the transition points improves control quality but at the price of energy efficiency. Figure 8.9n also shows that the destination of the returning water should not be selected on the basis of the origin of that water but rather should be based on temperature. This will reduce the upset caused in the plant utilities when a reactor switches from heating to cooling.

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**FIG. 8.9m**

If the control valves are provided with positioners, there will be three controllers in series in a cascade loop. It is recommended that all cascade masters be provided with an external reset from the measurement of their slave controller.
Figure 8.9o describes a reactor with a separate chilled water coil. This coil is inoperative until the cold water valve approaches full opening. When the valve position controller (VPC) detects that condition, it starts opening the chilled water valve. The resulting increased heat removal will cause the temperature cascade loop to close the cold water valve until it drops to the setting of the VPC.

Figure 8.9p serves the same purpose as the system depicted in Figure 8.9k, except that the designer placed the throttling valve for the coolant into the return flow path so that a separate back-pressure regulator would not be needed.
Pressure-Compensated Temperature Control

In a process where the reactor pressure is a function of batch temperature (e.g., the reactor pressure is essentially the vapor pressure of one of the major components in the reaction), this pressure may be sensed and used to speed up temperature control. In large polymerization reactors having low heat-transfer coefficients and large changes in heat evolution, the conventional temperature cascade loop is not fast enough. On the other hand, pressure measurement gives an almost instantaneous indication of changes in temperature.

Figure 8.9q illustrates the application of a pressure-compensated temperature control system to a reactor with both jacket and overhead condenser cooling (U.S. Patent No. 3,708,658, January 2, 1973, assigned to Byrd Hopkins of the Monsanto Company). This same approach can also be applied to reactors with jacket cooling only. Under steady-state conditions the reactor temperature \( T_m \) is on setpoint, set by the cam programmer, and therefore the calculated temperature \( T_c = T_m \).

When an upset occurs, the pressure transmitter (PT) will detect it first, causing the calculated temperature \( T_c = \sum \) to change as the AP part of the expression is changed. This will make the measurement of \( T_C-1 \) much faster than it otherwise would have been; it also allows the overhead condenser to start removing the excess heat even before the temperature transmitter (TT-1) is able to detect it. After each dynamic upset, the PI controller slowly returns the calculated temperature \( T_c \) to equal the measured temperature \( T_m \). This then automatically reestablishes the correct pressure-temperature relationship as the composition in the reactor changes.

The net result is the ability to operate the reactor at a much higher reaction rate, thereby obtaining higher productivity than was possible with temperature control alone.

TC-1 in Figure 8.9q would normally be provided with proportional and derivative control modes only. When TC-1 is on set point, the output signal returns to a value set by an adjustable internal bias. If the preferred means of cooling is through the jacket, then this bias will be set to a low value, but not to zero.

If there is only a single manipulated variable, the control system would be configured as in Figure 8.9r. The control modes are distributed between the measured variables so that integral action will act on the slowly responding temperature, while derivative action is applied to the more sensitive pressure.\(^{5}\)

The derivative time setting will be much shorter than the integral, because pressure responds faster than temperature. Integral action cannot be applied to the pressure measurement, because the pressure can vary even under constant temperature conditions (as a result of variation in feed composition or catalyst activity), and the intent here is to use the pressure loop only in the unsteady state.

Naturally, integral action is applied to the temperature measurement signal, because it is steady-state temperature...
that determines product quality, and integral action will ensure its return to set point.

**Temperature Controlled by Boiling Coolant**  Gas phase reactions (such as ammonia synthesis) are usually conducted at relatively high temperatures and pressures. Because of the competing reactions taking place in parallel with the main reactions, productivity and partial pressures are interrelated.

For example, in an ammonia synthesis process, a pressure increase is required to increase production when the ammonia concentration is low. On the other hand, a pressure decrease is required to increase production when the ammonia concentration is high. If inerts are also present, partial pressures are usually determined by detecting both the total pressure and the gas composition.

In such processes, which are both temperature- and pressure-sensitive, a very stable heat removal system is desired. If the reactor jacket is filled with boiling water, the rate of heat removal can vary without causing a change in the jacket temperature. Figure 8.9s illustrates such a cascade system. In order to increase the sensitivity of the loop, pressure is selected as the controlled variable for the slave controllers.

**Temperature Controlled by Feed Rate**  In a hydrocracking reactor (Figure 8.9t), product quality, catalyst life, and productivity are each a function of accurate temperature control. This combination would allow the reaction temperature to be controlled by manipulating the feed rate. If it is desirable to set production rate, and therefore feed rate independently of cooling capacity, the temperature can be controlled by throttling a diluent.

In Figure 8.9t the diluent is hydrogen, which is admitted under separate temperature controls into each zone. The introduction of hydrogen diluent lowers the reaction rate by reducing the reactant concentration and also by cooling.

The temperature of the batch at any one time is a function of the balance between the exothermic heat that is generated and the quantity of heat that is being removed by the coolant. If the two are not in balance, the batch temperature changes.

In the previously described control schemes, the exothermic heat was uncontrolled, and the cascade loop controlled the batch temperature by adjusting the coolant flow to match that load. In some of the fast and more stable processes, this configuration can be reversed, as shown in Figure 8.9u. Here the rate of cooling is fixed (at a relatively high rate) and the temperature controller (TRC-4) is adjusting the reagent flow (exothermic heat generation) to maintain a balance.

At the beginning of the batch the concentrations of precharged other reactants are high, the concentration of the product in the batch is low, and therefore the process gain is very high. As the reaction progresses, product concentration rises, unreacted reactant concentrations drop, and, as a consequence, the gain of the process also drops. By the end of the reaction, the process gain ($G_p$) can be reduced 10- or 100-fold.

As was shown in Figure 2.1x in Chapter 2, good control requires that the gain product of the loop be constant. Therefore, as $G_p$ drops it is necessary to increase the controller gain ($G_c$) in proportion. This is done by FY-1 in Figure 8.9u, which measures the total amount of reactant charged and increases the gain of TRC-4 as that total rises. The function of the reaction rate (FY-1) is nonlinear reaction-specific. TT-2 provides a feedforward signal so that if the coolant temperature would rise and thereby the cooling rate would drop off, this would also decrease the gain of TRC-4, thereby making it cause a smaller change in slave set point.

The set point of the slave (FRC-1) is limited by the low signal selector TY-4. It compares two limits to the set point of FRC-1, generated by TRC-4 and picks the lowest of the three signals. During start-up, safety is served by KT-4, which
8.9 Chemical Reactors: Basic Control Strategies

ramps the slave set point up at a safe rate to protect against overshooting. The slave (FRC-1) is preloaded to minimize start-up overshoot or transients.

The amount of preload is based on experience and serves to stabilize the loop more quickly than if the initial value of the FRC-1 output was zero. The main advantage of such control systems is that they minimize the batch reaction time and thereby maximize plant productivity.

Model-Based Temperature Control

When a reaction is highly exothermic and if runaway reactions can occur, the use of a model-based control system is justified. In order to develop the total model, three component models need to be developed:

1. **Equipment model**, describing the effect of heat-transfer media on reactor temperature ($T_r$)

2. **Kinetic model**, describing the effect of $T_r$ on the chemical reaction

3. **Calorimetric model**, describing the effect of reaction rate on $T_r$

When an exothermic reaction is taking place within the reactor, a feedback cascade loop such as that shown in Figure 8.9m is needed to provide stable temperature control by matching the rate of heat removal to the rate of heat generation. If the TRC in Figure 8.9m was tuned at a time when there was no reaction taking place within the reactor, it will not perform properly when exothermic heat is being generated.

If the gain of the master TRC is fixed, the loop might become unstable if the reaction is autocatalytic (does not require a catalyst). Model-based controls can improve on such performance. One such model-based approach is to use a heat-release rate estimator. The rate of heat release ($Q$ in Figure 8.9v) can be estimated on the basis of reactor

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temperature ($T_r$) and jacket-inlet temperature ($T_{jin}$) in accordance with:

$$Q = (T_r - K_1 T_{jin} + K_2)/K_2$$  \hspace{1cm} 8.9(6)$$

In exothermic reactions, $Q$ is the major load variation that tends to upset the stability of the temperature controls. Once this disturbance load is estimated, it is possible to feedforward that estimate to the slave set point as a bias. This bias relay is TY-2 in Figure 8.9v, and the feedforward bias (FF) it adds to the slave set point is a function of $Q$. The feedback master (TRC-1) naturally corrects the total effect on the jacket temperature set point and thereby overcomes the errors in the feedforward model.

The gain of the feedback master (TRC-1) is varied as a function of $Q$ [Gain = $f(Q)$]. As $Q$ increases, the process gain drops, and therefore the gain of this nonlinear adaptive-gain feedback controller is increased in order to keep the gain product for the loop constant.

**INITIAL HEAT-UP**

In most chemical reactions, a certain temperature must be reached in order to initiate the reaction. An ideal reactor temperature controller will permit rapid automatic heat-up to reaction temperature without overshoot and then accurately maintain that reaction temperature for several hours. This is a difficult goal to accomplish, because the dynamics of the controlled process will go through a substantial change as the heat load first drops to zero and then the cooling load gradually evolves when the reaction is started.

The master temperature controller is usually a three-mode one, tuned for the exothermic phase of the reaction cycle. It might have a 30% proportional band and a 5 minute setting for both integral and derivative. If such a controller were kept on automatic during heat-up, a substantial temperature overshoot would result (Figure 8.9w) because of windup. Therefore, the conventional PID controller must be supplemented with added features to provide it with the required start-up characteristics.

The added feature is either the “batch unit” or the “dual mode unit,” depending on the proportional band. If it is less than 50%, the batch unit will give good results, whereas if a
It is also necessary to provide a low limit (called preload) to the feedback signal; otherwise, the opposite of an overshoot would be experienced—an excessively sluggish approach to set point, as shown in Figure 8.9w by the “no preload” curve. If the PL setting did not prevent the feedback (F) from dropping too low at times of high error (such as at the beginning of heat-up), B could saturate at the low limit, keeping output (O) below zero even when the measurement has returned to set point. With preload, the controller output “O” will equal PL when the error is zero.

It can be seen from the above that a PID-type batch controller requires a total of five adjustments, because HL and PL must be set and the three control modes must be tuned. HL should be set at the maximum allowable jacket water temperature, which would then eliminate the need for a separate limit, such as the HIC in Figure 8.9l. The correct setting for PL is the master controller output at that time when reaction has started and a steady state has been reached between the generation and the removal of the heat of reaction. If, for example, the jacket water temperature during steady state is 90°F, this value could be selected as the preload setting, which will be the output of the master (and the set point of the slave) when the reaction temperature has been reached.

Actually, the PL setting should be a few degrees lower than this value, say 87 or 88°C, to allow for the contribution of the integral action of the controller from the time the proportional band is entered to the time where the set point is reached. This is illustrated in Figure 8.9y.

**Dual-Mode Controller**

The effectiveness of the batch unit, described earlier, is lost when the reactor requires a wide proportional band, say in excess

---

**The Batch Unit**

Without the batch unit, the PI controller illustrated in Figure 8.9x would receive a feedback signal (F) equaling the output (O). Therefore, whenever there is an error (E), the output signal is driven continuously by the positive feedback through I (a first-order lag, having a time constant I) until B reaches the saturation limit. Once in this saturated state (the reset is wound up), the output signal “O” will equal “B” even if the error has returned to zero. This is the reason that in Figure 8.9w the temperature keeps rising even after it has reached set point (SP = M, E = O).

Without the batch unit, therefore, control action cannot begin until an equal and opposite area of error is experienced. This is why reset windup always results in overshoot and why this windup must be eliminated by the addition of the batch unit shown in Figure 8.9x.

Under normal operation, the output to the valve is below the high limit. Therefore, G is positive and the amplifier drives D upward, which causes O to be less than C. In this state, the low selector selects O as the feedback signal, and the controller behaves as a conventional PI controller.

When O exceeds HL, the amplifier drives down D, C, F, and B and thereby limits O from exceeding the HL setting.
of 50%. With a wide band (as shown in Figure 8.9y) reset action would begin much earlier, which would lengthen the heat-up time. In such a case, the dual-mode unit (Figure 8.9z) is the proper selection.

In the dual-mode unit, the preload is estimated as in the case of the batch unit, but it is not reduced for integral correction. It is not lowered from $90^\circ F$ to $87$ or $88^\circ F$ in our example, because in this case reset does not begin until the error is zero.

The sequence of operation is as follows:

1. Full heating is applied until the reactor is within 1 or 2% of its set-point temperature. This margin is set by the minimum error setting ($Em$). During this state SS-1 and SS-2 are in position “A.”
2. When $E$ drops to $Em$, time delays TD-1 and -2 are started, and full cooling is applied to the reactor for a minute or so to remove the thermal inertia of the heat-up phase. When TD-1 times out the period required for full cooling, SS-1 switches to position “B” and the PID controller output is sent to the slave as set point. This output is fixed at the preload (PL) setting, which corresponds to the steady-state jacket temperature (estimated in our example as $90^\circ F$).
3. When the error and its rate of change are both zero, estimated by TD-2, this time delay will switch SS-2 to position “B.” This switching also transfers the PID loop from manual to automatic, with its external feedback loop closed.

If properly tuned, the dual-mode unit is the best possible controller, because by definition, optimal switching is unmatched in the unsteady state by any other technique. On the other hand, this loop requires seven settings. Three of these—$P$, $I$, and $D$—pertain only to the steady state of the process; the other four—PL, $Em$, TD-1, and TD-2—will determine start-up performance. The effect of these adjustments is self-evident:

- $Em$ should be increased in case of overshoot and lowered if undershoot is experienced.
- PL has the same effect as in Figure 8.9w.
- TD-1, if set too long, will bring the temperature down after the set point is reached.
- TD-2 is not very critical.

Figure 8.9aa illustrates that from the start-up performance of the reactor, it can be determined which setting needs adjustment.

**Rate of Temperature Rise Constraint**

In highly unstable, accident-prone reactors that have a history of runaway reactions, an added level of protection can be provided, based on the permissible rate of temperature rise.
The actual rate of temperature rise in units of safety backup. The protection is provided by first calculating during heat-up. This is usually superimposed on the previously discussed control systems on a selective basis as a safety backup. The protection is provided by first calculating the actual rate of temperature rise in units of °F/min and then sending it as the measurement signal to the RRIC controller in Figure 8.9bb.

The set point of this controller is programmed as a function of the heat-up state. Toward the end of the heat-up period, when the reaction temperature has been almost reached (the error is near zero), the value of the permissible rate of rise is set to be much lower than at the beginning of heat-up. Doing so prevents the process from building up a thermal inertia as it approaches the region of potential instability.

Model-Based Heat-Up Control

Model-based heat-up algorithms can be configured in two blocks, the “predictor block” and the “corrector block.” The predictor block is a process model of the reactor and serves to determine the amount of heat input required \( Q_h \) at any point in the batch heat-up cycle, so as to minimize the heat-up period while eliminating overshoot.

The corrector block serves to refine the model by correcting the constants and calculation parameters in the process model by comparing the model to the actual process. Figure 8.9cc illustrates the operation of such a model-based heat-up control system.

The variable manipulated by the “predictor” is the heat input rate \( Q_h \), which initially is set to maximum, similar to the action of the dual-mode control algorithm in Figure 8.9z. The difference with the dual-mode strategy is that here (in Figure 8.9cc), as the reactor temperature approaches its target set point, the manipulated variable is not switched to cooling but to a minimum rate of heating \( Q_h \). The switching from \( Q_h \) max to \( Q_h \) min occurs when the steady-state reactor model indicates that with the amount of heat already introduced, once the reactor reaches its new steady state, its temperature will match the set point.

**Adaptation**

The nature of the batch reactor process is such that it never reaches a steady state. The process is in a continuous state of transformation and change; therefore, the control system parameters should also be in a continuous state of change. Some of these changes are rather drastic; for example, when the system switches from heating to cooling. When such changes occur, the controller parameters must be changed immediately and equally drastically, which is usually done by “programmed adaptation,” meaning that the PID algorithm settings for the various stages of the reaction cycle are stored in a table and are recalled as needed.

Adaptation of tuning constants can also occur slowly using various adaptive algorithms (Chapter 2) as a function of various measurements, such as, for example, the rising of the level (mass) in the reactor or the reduction in the exothermic heat release as the reaction rate drops off. Adaptation can also be based on various rules of thumb, such as utilizing the rate of temperature rise during heating up the reactor to predict the reactor time constants, which in turn can be used to tune the temperature controller.

**Initialization, Preload, and Feedforward**

When the transition from heat-up to the reaction phase takes place, it is important that the temperature controller be properly “initialized” (placed into the correct initial state). That means that the controller must be provided with the proper tuning constants and bias (output signal at zero error) that will make the transition smooth. This initialization of the controller is often called preloading. As was shown in Figure 8.9w, the use of the right amount of preload is essential to obtain maximum heat-up rate without overshoot.

In a batch process, where the same product is made in consecutive batches, preload is often determined empirically. This means that if in batch #19 the transition to reaction phase was stable and the controller output at the beginning of that phase was 52%, it is a fairly safe assumption that in batch #20 the same 52% preload will also provide stable transition. In some reactor heat-up applications the preload can also be predicted on the basis of the rate of temperature rise during heat-up.

Feedforward features can also be incorporated into the overall control system. For example, in Figure 8.9n one could feedforward a change in the set point in the master directly into a change in the set point of the slave, if a bias unit was inserted to detect the master set point and to generate the correct amount of change based on that measurement. By such feedforward action, the integral-mode offset error, caused by set point change, can be reduced.

This is a good feature, because even if the control algorithm is so configured that the proportional and derivative modes act only on the measurement (Section 2.3 in Chapter 2), the integral mode usually acts on the total error, and therefore it is responsive to set-point changes.
In exothermic reactions, one of the critical safety constraints is coolant availability. That limitation is automatically configured into the control loop of a continuous reactor, illustrated in Figure 8.9dd. Here, the optimizing controller (OIC) detects the opening of the coolant valve, and if it is less than 90% open, it admits more feed by increasing the set point of the FRC. Thus, production rate is always maximized, but only within the safe availability of the coolant. When the...
cooler valve opens beyond 90%, production rate is lowered so that the reactor will never be allowed to run out of coolant.

Another safety feature shown in Figure 8.9dd is the reaction failure alarm loop (RA). This loop compares the reactant charging rate to the reactor with the heat removal rate from the reactor and actuates an alarm if the charging rate is in substantial excess.

In Figure 8.9dd, the valve position controller (OIC) maintains the feed rate as high as the cooling system can handle. This results in a variable production rate, because changes in coolant temperature or variations in the amount of fouling of the heat-transfer areas will change production.

The dynamic response of the reactor temperature to changes in reactant flow is not favorable. A change in feed flow must change the reactant concentration before a change in reaction rate (and therefore in heat evolution) can change the reaction temperature.

Reactant concentration adds a large secondary lag to the valve position control loop. This necessitates the use of a three-mode controller,\(^5\) which in turn limits the application to stable loops only. The slower the OIC loop (the longer its lag), the lower should be the set point of OIC in Figure 8.9dd, to provide the required margin for stability.

**Maximizing Batch Reactor Production** In a batch reactor, production rate increase with temperature, and therefore production can be maximized by maximizing temperature. This can be accomplished by a valve position controller that raises the batch temperature set point whenever the coolant valve is less than 90% open.

Unfortunately, the dynamic characteristics of this loop are also undesirable because of the inverse response of the loop.\(^5\) When the coolant valve opens to more than 90%, the VPC will lower the set point of the temperature controller. This will temporarily increase the demand for coolant, although once the excess sensitive heat is removed, it will lower it. The longer the dead time introduced by this inverse response, the large the margin needed for safety and stability, and hence the lower the set point of the VPC.

When the concentration time constant is equal to or larger than the thermal time constant, temperature control through feed flow manipulation is no longer practical.\(^5\) In such reactors, only the manipulation of the coolant will give stable operation. The strategy of valve position control is still useful in such applications, but in these cases it must manipulate supplementary cooling.

**Heat Release Control**

By multiplying the jacket circulation rate by the difference in temperature between inlet and outlet, it is possible to determine the amount of heat released or taken up by the reaction. The reaction is in an endothermic state when the value of \(Q\) in Figure 8.9ee is negative, and it is in an exothermic phase when \(Q\) is positive.

Under steady-state conditions, at high circulation rates the difference between \(T_o\) and \(T_i\) is about 5°F or less. The

**FIG. 8.9dd**
The reaction rate in this continuous reactor is continuously matched to the full available capacity of the cooling system.

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method of reading this $\Delta T$ is critical, because during load changes, dynamic compensation is needed. As more cooling is requested, the jacket inlet temperature will drop, and the jacket outlet temperature will stay unaffected until the jacket contents are displaced. If $Q$ is to be used for control purposes, such dynamic errors must be removed by compensation.

This is accomplished by delaying the jacket inlet temperature into the $\Delta T$ transmitter by a time equal to the delay through the jacket. In Figure 8.9ee, this is accomplished by simulating the jacket using a length of tubing whose dead time is adjustable by changing the flow rate through it. A similar result can be obtained by placing an inverse derivative relay, set for a jacket displacement time of, say, 30 secs, into the transmitter output signal; the desired goal can also be reached by electronically simulating the transportation lag in digital systems.

The correct selection of the $\Delta T$ Transmitter is very important, because in order to be able to detect $Q$ within a $\pm 1\%$ error, the $\Delta T$ must be detected within $\pm 0.2^\circ F (\pm 0.01^\circ C)$. This can be accomplished only by using the best quality RTD-type transmitters that can provide spans, which are as narrow as $-5$ to $0$ to $+5^\circ F$. When the absolute value of the jacket temperature can vary from, say, 50 to 250$^\circ F$ throughout the reaction cycle, it will also be necessary to correct the calibration of the $\Delta T$ transmitter as the absolute temperature changes.

Once the value of $Q$ is accurately determined, it can be used for many purposes. The instantaneous value of $Q$ signals the rate of heat removal or addition, and the time integral of $Q$ gives the total heat that has been added or removed. A change in the value of $\Delta T$ under standard conditions can signal fouling and the need for cleaning the reactor heat-transfer surfaces.

**Stripping and Refluxing Controls**

During stripping or refluxing phases, the reactor might be controlled on the basis of heat input ($Q$), because refluxing tends to be done at constant temperature, and the increase in temperature during stripping is usually too small for control purposes. Therefore, the system would be switched automatically from temperature control (Figure 8.9ee) to heat input control (Figure 8.9ff) whenever the reactor enters a stripping or refluxing phase.

The heat input during refluxing is usually set to be sufficient to maintain a state of slow boiling. During stripping, the heat input is usually set to complete the stripping in some empirically established time period; for example, an hour or two.

The time integral of $Q$ represents the total reaction heat, which is an indicator of product concentration or percentage conversion. It can be used to introduce additives at predetermined conversions and to determine reaction endpoint. Through these automated steps, the need for taking grab samples can be eliminated. This in turn will result in reduced overall cycle time and, therefore, in increased production rate. The control of vacuum strippers will be discussed in the Pressure Control subsection.
In order to keep the reaction rate from dropping off, the reaction temperature has to be increased (it is a nonisothermal batch reaction). Changing the operation of a batch reactor from a constant reaction temperature to a variable reaction temperature mode (Figure 8.9b) is not an easy proposition to carry out.\(^7\) One of the disadvantages of a nonisothermal operation is that the formed products might change as the reaction temperature changes. Yet, in some processes, the economic advantages outweigh the required complexities in modeling and control.

One possible configuration is to use the reactor shown in Figure 8.9v, in which the reactant is charged at constant rate, and use the heat-release estimator in that figure to modify the set point of TRC-1 in such a way that the reaction rate will be held constant. For some reactions the temperature settings required to provide constant reaction rates can be calculated, while for more complete reactions it is necessary to determine them experimentally.

Figure 8.9hh shows a cascade configuration in which the reaction rate controller (XRC-4) is the master, and it adjusts the batch temperature (TRC-1) set point so as to keep the reaction rate constant. The measurement to the reaction rate controller can come from a heat-release estimator (Figure 8.9hh) or from the actual measurement of the exothermic heat release ($Q$ in Figures 8.9ee and 8.9ff).

**ENDPOINT DETECTION**

The reliable determination of batch cycle endpoint is important for two reasons:

1. One way to increase plant productivity is to reduce batch cycle time.

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**FIG. 8.9gg**

The batch reaction time is a function of the reaction rate within the reactor. If constant rate is maintained, the batch reaction time can be drastically reduced.\(^7\)

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**FIG. 8.9hh**

Nonisothermal (variable reaction temperature) batch reactor control can shorten the batch cycle time by keeping the reaction rate constant.
2. If the reaction continues beyond the optimal endpoint, the process yield can be reduced due to side reactions.

In batch reactors, if the completion of the reaction cannot be reliably detected, it is common practice to terminate reactant flow on the basis of total charge. This can be based on flow or weight measurements. In other reactions, the endpoint is accompanied by the fall or rise of the reaction pressure. This change in pressure can be used to have a pressure switch or pressure controller to shut off the reactant flow (Figure 8.9ii). In still other reactions, the total reaction heat signals the end-point, as was illustrated in Figures 8.9ee and 8.9ff.

**pH Endpoint** If some analytical property, for example, pH, is used in detecting the endpoint, it is important to realize that during most of the reaction the measurement will be away from set point. Therefore, integral action must not be used, because it would saturate and overshoot would result. This is one of the few processes in which the proper selection of control modes is a proportional plus derivative with zero bias, modulating an equal-percentage reagent valve.

If the derivative time is set correctly, the valve will shut quickly as the desired endpoint pH is reached. With excessively long derivative time setting, the valve will close prematurely, but this is not harmful because it will reopen as long as the pH is away from the set point.

For the measurement of pH, conductivity, and resistivity, on-line analyzers are available. The pH probe shown in Figure 8.9jj can be used for on-line measurement in a glass-lined reactor. When maintenance or recalibration is needed or when the reactor is empty, the probe can be lifted by a pneumatic actuator and immersed into buffer a solution to protect it from getting dry.

In some cases it might be preferred to keep the pH sensor outside the reactor and bring a sample of the reactor contents to it. Figure 8.9kk illustrates such a design configuration.

In other installations, such as in fermenters, the preference is for the use of side entry, retractable pH probes. These are illustrated in Figure 8.9ll. Other pH probe designs are described in Section 8.48 in Chapter 8 of the first volume of this handbook.

If one reactant is charged to the reactor in substantial excess to the others, this will usually guarantee the complete
conversion of the other reactants, and endpoint control is not required, unless the product is also used as the recycle stream. If the reactants are fed to the reactor in the same proportion in which they react, endpoint control is required. If the reactants and product are all in the same phase (all liquids or all gases), the endpoint is usually determined through the use of an analyzer, such as the pH unit.

On the other hand, when the reactants are in different phases, the endpoint can be controlled on the basis of level or pressure. As was shown in Figure 8.9ii, if the product is

**FIG. 8.9kk**
The design components of a chemical reactor sampling system.

**FIG. 8.9ll**
Retractable, side-entry probe designs.
Control and Optimization of Unit Operations

In continuous reactors it is usually desirable to provide a constant residence time for the reaction. If the production rate (inflow to the reactor) varies, the most convenient way of keeping the residence time constant is to maintain a constant ratio between reactor volume \( V \) and reactor outflow \( F \). The ratio \( V/F \) is the residence time, where the volume \( V \) can be approximated by a level measurement, and \( F \) can be measured as outflow. Figure 8.9nn illustrates a residence time control system using this approach.

All reactions with a liquid product can benefit from residence time control. This requires the combination of the features in the control system shown in Figures 8.9ii and 8.9mm, resulting in Figure 8.9nn.

**Analyzers for Endpoint Detection**

Probe-type analyzers do not require sampling systems, because the analyzer itself is inserted directly into the process. In the case of chemical reactors, analyzers can be inserted into either the reactor vessel or the discharge piping. Regardless of their locations, probes will give accurate readings only when clean. Therefore, automatic cleaning of such on-stream analyzer probes is a good idea.

Figure 8.9oo illustrates a flow-through, brush-type probe cleaner assembly that is provided with a sight glass, allowing visual observation of the cleanliness of the probe. Such units are also available with spray nozzle attachments for water or chemical cleaning of the electrode.

One common method of endpoint detection is the measurement of viscosity either by monitoring the torque on the agitator or by using viscosity sensors. Such a viscometer can be inserted in pipelines or directly into the reactor. It is essential that they be located in a representative area and be temperature-compensated.

In reactions in which the endpoint can be correlated to density, some of the more sensitive and less maintenance-prone densitometers can also be considered (Chapter 6 in Volume 1). The radiation-type design tends to meet these requirements if pipe line installation is acceptable. These
units can operate with a minimum span of 0.05 specific gravity unit and an error of ±1% of that span, or an absolute error of ±0.0005 specific gravity.

In a number of reactions, refractometers (Section 8.52 in Chapter 8 in Volume 1) have been found to be acceptable solutions to endpoint detection. In many installations, they are used as laboratory devices: A grab sample is brought to the bench analyzer. In other installations, they are used on-stream. When the refractive index (RI) analyzer is on-stream, human error tends to be reduced and analysis time is shortened, with a corresponding increase in productivity.

On the other hand, an on-stream installation requires more maintenance attention, because the RI measurement must be made to high precision, such as ±0.0005 RI units. This requires accurate temperature compensation, plus protection of the heat-sensitive photocells through cooling, which are time-consuming and expensive to replace when ruined by high temperature.

If the endpoint of a reaction is detectable by such measurements as a change in color or opacity, or the concentration of suspended solids, the self-cleaning probe shown in Figure 8.9pp can be considered. Here, the reciprocating piston in the inner cavity of the probe not only serves to clean the optical surfaces with its wiping seals but also guarantees the replacement of the sample in the sampling chamber by fresh material upon each stroking of the piston. The temperature considerations and limitations mentioned in connection with the RI analyzer also apply here.

If the endpoint of the reaction is detectable by infrared (IR) beam attenuation, such analyzers can also be put on-stream or can be inserted in pipelines and will detect the attenuated total reflectance of the IR beam. This type of analyzer is particularly suitable for the detection of water in hydrocarbons.

In addition to the analyzers discussed above a wide variety of computer-supported fiber-optic probes (FOPs) are also available for on-line analysis. Other analyzers, such as the gas chromatograph (GC), the high-pressure liquid chromatograph (HPLC), and the mass spectrometer (MS), are also powerful analytical tools and are available both as laboratory or on-line devices. For an in-depth discussion of all the analyzers available, refer to Chapter 8 in the Process Measurement and Analysis volume of this handbook.

**Product Quality Control**

Measuring the composition of the contents of a chemical reactor is a method that can help maintain product quality in continuous reactors and can detect endpoints in batch reactors. The analyzers are limited by sampling difficulties, by the intermittent nature of some models, and by incomplete mixing within the reactor. Because sampling and sample preparation result in dead time, sampling time should be minimized or eliminated altogether; this can be done by placing the sensor directly into the reactor, which results in tight control and fast response.

Because analyzers in general are low-reliability and high-maintenance devices, most users are reluctant to close an automatic loop around them. The main concern is that the failure of the analyzer might cause a hazardous condition by driving the reactor into an unsafe state. As shown in Figure 8.9qq, this concern can be alleviated by the use of
In case of a “downscale” failure of one of the analyzers, the backup unit takes over automatically. An “upscale” failure in this arrangement would shut down the reactor, providing safety at the cost of production.

If neither an accident nor an interruption of production can be tolerated, three analyzers are required, arranged in a “voting” configuration. In such an arrangement, if one of the sensors disagrees with the others, it is automatically disregarded and the “majority view” is used for closed-loop control. Maintenance and recalibration of the defective sensor are then initiated. Another solution is to use a median selector in combination with the three analyzers.

**PRESSURE CONTROL**

In gas phase reactions, in oxidation and hydrogenation reactions, or in high-pressure polymerization, the reaction rate is also a function of pressure. If, in a batch reaction, the process gas is completely absorbed, the controls in Figure 8.9rr would apply. Here, the concentration of process gas in the reactants is related to the partial pressure of the process gas in the vapor space. Therefore, pressure control results in the control of reaction rate. This loop is fast and easily controlled.
outlet line. Reactor pressure is sensed, and the overflow from the reactor is throttled to maintain the desired operating pressure. Process gas feed and process liquid feed streams are on flow control.

All these illustrations are simplified. For instance, it may be desirable to place one of the flow controllers on ratio in order to maintain a constant relationship between feed streams. If the reaction is hazardous, and there is the possibility of an explosion in the reactor (for example, oxidation of hydrocarbons), it may be desirable to add safety devices, such as a high-pressure switch, to stop the feed to the reactor automatically.

If one of the reactants differs in phase from both the other reactants and the product, inventory control can be applied (Figure 8.9ii). In the case of gaseous products, the reactor level is controlled by modulating the liquid reactant; with liquid products, the gaseous reactant is modulated to keep reactor pressure constant. A purge is needed in both cases to rid the reactors of inerts either in the liquid or in the gas phase.

Vacuum Control

Some reactions must be conducted under vacuum. The vacuum source is frequently a steam jet-type ejector. Such units are essentially venturi nozzles with very little turndown. This constant-capacity vacuum source is frequently matched to the variable capacity reactor by wasting the excess capacity of the ejector. This is done by creating an artificial load through the admission of ambient air.

When the vapor generation rate in the reactor is low, the steam jet is still operating at full capacity, sucking in and ejecting ambient air. The corresponding waste of steam can be substantially reduced if the system shown in Figure 8.9uu is applied. Here, a low-load condition is detected by the fact that the air valve is more than 50% open, and this automatically transfers the system to the “small” jet. A later increase in load is detected as the air valve closes (at around 5%), at which point the system is switched back to the high-capacity jet.

Vacuum Stripping

When stripping is done by direct steam injection, the controls shown in Figure 8.9ff are not applicable. The controls of a vacuum stripper serve to remove the solvent and the unreacted monomers from the batch in minimum time but without causing foaming.

The steam addition to the stripper (FIC in Figure 8.9vv) is usually programmed as a function of time (FY). As the batch gets more concentrated with polymer products and leaner in monomers or solvent, the more likely it is to foam, and therefore the rate of addition is reduced. This is usually controlled by hardware or a software program (FY) that relates steam flow to time. This relationship is adjustable, and if foaming is experienced during a stripping cycle (LSH in Figure 8.9vv), the steam rate curve programmed in FY can be lowered to avoid the repetition of foaming in the next batch.

The temperature of the batch is a function of (1) the balance between the amount of heat introduced by the steam and (2) the amount of cooling caused by the vaporization of the monomers. The vacuum level in the stripper is therefore controlled by temperature. This guarantees that the vacuum is low enough so that all the solvents and monomers are removed, but that it does not drop too low, to the point where the stripping steam would condense and would not be removed by the vacuum but would cause dilution of the batch.

REACTOR SAFETY

For a more detailed discussion of plant and control system safety, the reader is referred to Sections 4.11 and 5.8 of this volume. Here, only some of the “reactor-specific” safety concerns will be considered.

Safety problems can arise in chemical reactors as a result of many causes, including equipment failure, human error, loss of utilities, or instrument failures. Depending on the nature of the problem, the proper response can be to “hold” the reaction sequence until the problem has been cleared or to initiate an orderly emergency shutdown sequence. Such actions can be taken manually or automatically.

After a “hold” or “emergency” condition has been cleared, an orderly sequence of transitional logic is required to return the reactor to normal operation. It makes no difference whether the emergency was caused by a pump or valve failure or whether the reactor was put on hold to allow for
manual sampling and laboratory analysis of the product: A return sequence is still required. The reentry logic determines the process state when the interruption occurred and then decides whether to return to that process state or the previous one in order to reestablish the conditions that existed at the time of the interruption.

A reactor control system should provide the following features:

- The ability to maximize production
- The ability to minimize shutdowns
- The ability to maximize on-line availability of the reactor
- The ability to minimize the variations in utility and raw material demand
- The ability to provide smooth operation in terms of constant conversion, yield, and product distribution
- Easy start-up and shutdown

However, the overriding, primary design objective is that the reactor must be safe for both the operating personnel and the environment.

Safety is guaranteed by monitoring both the potential causes of emergencies and their consequences. If the cause is detected and responded to in time, the symptoms of an emergency will never develop. It is for this reason that the monitoring of potential causes of safety hazards is preferred.

Some of the potential causes of emergencies and the methods used to sense them are listed in Table 8.9ww.

Each of the above failures can actuate an annunciator point to warn the operator and can also initiate a hold or shutdown sequence, if the condition cannot be corrected automatically. Automatic correction will require different responses in each case.

---

**TABLE 8.9ww**

Methods of Detection and Potential Causes of Reactor Emergencies

<table>
<thead>
<tr>
<th>Causes</th>
<th>Methods of Detection</th>
</tr>
</thead>
<tbody>
<tr>
<td>Electric power failure</td>
<td>Volt or watt meters</td>
</tr>
<tr>
<td>Instrument air failure</td>
<td>Pressure switch</td>
</tr>
<tr>
<td>Coolant failure</td>
<td>Pressure or flow switches</td>
</tr>
<tr>
<td>Reactant flow failure</td>
<td>Pressure or flow switches</td>
</tr>
<tr>
<td>Catalyst flow failure</td>
<td>Pressure or flow switches</td>
</tr>
<tr>
<td>Loss of vacuum</td>
<td>Vacuum switch</td>
</tr>
<tr>
<td>Agitator failure</td>
<td>Torque or RPM detector</td>
</tr>
<tr>
<td>Pump failure</td>
<td>Pressure or flow switch</td>
</tr>
<tr>
<td>Valve failure or mispositioning</td>
<td>Position-sensing limit switches</td>
</tr>
<tr>
<td>Measuring device failures</td>
<td>Validity checks</td>
</tr>
</tbody>
</table>
emergency shutdown is higher. For example, if, in a potentially discussed failure causes, but here the probability of an abnormal process variable condition much as it did to the previous critical discussion.

The control system will respond to the symptom of an abnormal condition. This might involve temperature, pressure, composition, or other conditions listed in Table 8.9xx.

<table>
<thead>
<tr>
<th>Symptom</th>
<th>Detectors</th>
</tr>
</thead>
<tbody>
<tr>
<td>High reactor temperature</td>
<td>High temperature switch</td>
</tr>
<tr>
<td>High jacket temperature</td>
<td>High temperature switch</td>
</tr>
<tr>
<td>Low jacket temperature</td>
<td>Low temperature switch</td>
</tr>
<tr>
<td>High reaction rate</td>
<td>High heat release detector</td>
</tr>
<tr>
<td>Reaction failure</td>
<td>Low heat release detector</td>
</tr>
<tr>
<td>High rate of temperature rise</td>
<td>Rate of rise detector</td>
</tr>
<tr>
<td>High pressure</td>
<td>High-pressure switch and relief valve</td>
</tr>
<tr>
<td>Abnormal level</td>
<td>Level and weight switches</td>
</tr>
<tr>
<td>Abnormal composition</td>
<td>On-stream analyzers</td>
</tr>
</tbody>
</table>

For example, in case of electric power failure, a diesel generator or steam turbine might be started. If instrument air failure is detected, an alternate compressor might be started, or an alternate gas, such as dry nitrogen, might be used. In addition, the safe failure positions for all control valves must be predetermined, as in Figure 8.9m. In case of loss of coolant, an alternate source of coolant might be used, or the type of automatic constraint control that was shown in Figure 8.9dd might be applied.

It is also desirable to provide intelligent human–machine interfaces that will protect against unsafe operator actions. The simplest example of such limits is to prevent the operator from moving set points outside safe limits.

If reactant or catalyst flow fails, automatic ratio loops (Figure 8.9dd) can cut back the flow rates or related streams; alternatively, if there are alternate storage tanks from which the reactant can be drawn, these tanks might be accessed. A loss of vacuum can also be corrected by switching to an alternate vacuum source if such redundancy is economically justifiable.

Critical pumps and valves can be provided with spare backup units, and agitators can be furnished with multiple drives. Failed sensors and defective instruments can be automatically replaced by redundant and voting systems.

If the cause of an emergency goes undetected, eventually it will affect the operating conditions of the reactor. These symptoms might involve temperature, pressure, composition, or other conditions listed in Table 8.9xx.

Runaway Reactions

The control system will respond to the symptom of an abnormal condition. This might involve temperature, pressure, composition, or other conditions listed in Table 8.9xx.

Glass Lining Protection

Some of the chemical reactors are glass-lined to resist corrosion or to protect against contamination or discoloring of the product. When a reactor is glass-lined, the integrity of the lining can be damaged, because the thermal expansion characteristics of glass and metal are different. Therefore, it is necessary to provide control systems that serve to protect the lining against cracking (Figure 8.9yy) and that are able to detect if such damage has occurred (Figure 8.9zz).

The integrity of various glass-lining materials is guaranteed for different temperature differentials between the metal and the glass. The lining protection controls serve to keep the difference between the temperature of the lining (batch temperature detected by TT-1 in Figure 8.9yy) and the temperature of the metal (jacket temperature detected by TT-2) under the guaranteed limit.

Figure 8.9yy illustrates the protection loop for a situation in which the maximum differential is 100°. The goal of the controls is to keep the set point of the slave (TRC-2) within 100° of the batch temperature (TT-1). The purpose of TY-3 and TY-4 is to make sure that the slave set point does not drop too low. Therefore, TY-3 subtracts 100° from the TT-1 signal, while TY-4 selects the higher between that signal and the master (TRC-1) output. Similarly, the purpose of TY-5 and TY-6 is to prevent the slave set point from rising too high. TY-5 adds 100° to the TT-1 signal, while
TY-6 selects the lower between that signal and the TRC-1 output.

Figure 8.9zz shows the control system used to monitor the integrity of the glass lining, when the batch is electrically conductive. The fault test is made by inserting a probe into the batch and checking the resistance between that and the metallic tank wall. If the glass lining is damaged, the resistance drops as the metal is exposed to the conductive batch. The fault monitor shown in Figure 8.9zz is designed for the scanning of ten glass-lined reactors.

**Multiple Sensors**

In connection with the topic of increasing the reliability of composition analyzers, we have already seen some options on using redundant measurements in our control systems. Figure 8.9qq described one possible option, where a pair of redundant analyzers in combination with a high signal selector can increase reliability.

Multiple temperature sensors can also be used for safety or maintenance reasons. In a fixed-bed reactor, for example, in which the location of the maximum temperature might shift as a function of flow rate or catalyst age, multiple sensors would be installed and the highest reading selected for control, as shown in Figure 8.9aaa.

On the other hand, if the reason for the use of multiple sensors is to increase the reliability of the measurement, the use of median selectors is the proper choice (Figure 8.9bbb). A median selector rejects both the highest and the lowest signals and transmits the third. This type of redundancy protects against the consequences of sensor misoperation, while
also filtering out noise and transients that are not common to two of the signals.

Another method of increasing sensor reliability is the use of voting systems. These also consist of at least three sensors. Reliability is gained, as the voting system disregards any measurement that disagrees with the majority view.

Instrument Reliability

A risk analysis requires data on the failure rate for the components of the control system. Such data has been collected from user reports; the findings of one such survey are shown in Table 8.9ccc. When the mean time between failure (MTBF) of the individual instrument components has been

![FIG. 8.9zz]
*The illustrated fault finder signals if the glass lining of a reactor is damaged. (Courtesy of Factory Mechanical Systems.)*

![FIG. 8.9aaa]
*This method can be used to control the highest temperature in a fixed-bed reactor.*

![FIG. 8.9bbb]
*Median selector rejects both the highest and the lowest signals and can be the logical choice in some applications.*
Control and Optimization of Unit Operations

estimated, the MTBF of the various control loops can be established.\(^9\)

An example of predicting the MTBF of a system, based on the MTBF of its components, is illustrated in Table 8.9ddd. Once the MTBF of each loop has been established, the reliability of the whole reactor control system can be increased by increasing the MTBF of the loops that pose the highest risks. This usually is done through the use of self-diagnostics, preventive maintenance, backup devices, and, in critical instances, the technique of “voting,” which was described in connection with Figure 8.9bbb.

While risk analysis is complex and time-consuming (see Sections 4.11 and 5.8), a plant can be designed for a particular level of safety, just as it can be designed for a particular level of production. The reliability of the result is as good as the data used in the analysis. For this reason, data collected by users, testing laboratories, or insurance companies

should be used, instead of manufacturers’ estimates. For instrument reliability and performance data, good sources are the International Associations of Instrument Users, SIREP and WIB.

CONCLUSIONS

As was discussed in the previous paragraphs, the goals of good chemical reactor control and optimization include the following:

- Precise recipe charging
- Minimum heat-up time without temperature overshoot
- Maximized reaction rate within limits of coolant availability
- Accurate and fast endpoint determination

These goals can be met by various levels of automation. As the requirements for safety and optimized production are increased, higher and higher levels of automation become necessary. The trend is towards model-based anticipation and towards the integration of the individual control loops into integrated multivariable reactor controller systems.\(^13\)

References


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### TABLE 8.9ccc

<table>
<thead>
<tr>
<th>Variable</th>
<th>Instrument</th>
<th>Mean Time between Failures</th>
</tr>
</thead>
<tbody>
<tr>
<td>Level</td>
<td>Bubbler</td>
<td>1–2 yrs.</td>
</tr>
<tr>
<td></td>
<td>d/p transmitter</td>
<td>1–5 yrs.</td>
</tr>
<tr>
<td></td>
<td>Float and cable</td>
<td>0.2–2 yrs.</td>
</tr>
<tr>
<td></td>
<td>Optical</td>
<td>0.1–5 yrs.</td>
</tr>
<tr>
<td>Flow</td>
<td>Flume and weir</td>
<td>0.5–5 yrs.</td>
</tr>
<tr>
<td></td>
<td>Venturi, etc.</td>
<td>2 mo.–5 yr.</td>
</tr>
<tr>
<td></td>
<td>Propellers</td>
<td>1 mo.–1 yr.</td>
</tr>
<tr>
<td></td>
<td>Positive displacement</td>
<td>1 mo.–1 yr.</td>
</tr>
<tr>
<td></td>
<td>Magnetic</td>
<td>0.5–10 yrs.</td>
</tr>
<tr>
<td>Density</td>
<td>Nuclear</td>
<td>1–3 yrs.</td>
</tr>
<tr>
<td></td>
<td>Mechanical</td>
<td>1–6 mos.</td>
</tr>
<tr>
<td>Analysis</td>
<td>pH and ORP</td>
<td>1–4 mos.</td>
</tr>
<tr>
<td></td>
<td>Dissolved O(_2)</td>
<td>1–9 mos.</td>
</tr>
<tr>
<td></td>
<td>Turbidity</td>
<td>1–6 mos.</td>
</tr>
<tr>
<td></td>
<td>Conductivity</td>
<td>1–4 mos.</td>
</tr>
<tr>
<td></td>
<td>Chlorine gas</td>
<td>0.5–1 yr.</td>
</tr>
<tr>
<td></td>
<td>Explosive gas</td>
<td>0.2–1 yr.</td>
</tr>
<tr>
<td></td>
<td>TOC</td>
<td>0.1–1 mo.</td>
</tr>
<tr>
<td>Miscellaneous</td>
<td>Temperature</td>
<td>0.5–2 yrs.</td>
</tr>
<tr>
<td></td>
<td>Pressure</td>
<td>0.1–5 yrs.</td>
</tr>
<tr>
<td></td>
<td>Speed</td>
<td>0.6–5 yrs.</td>
</tr>
<tr>
<td></td>
<td>Weight</td>
<td>0.6–2 yrs.</td>
</tr>
<tr>
<td></td>
<td>Position</td>
<td>0.1–1 yr.</td>
</tr>
<tr>
<td></td>
<td>Sampling</td>
<td>0.1–1 yr.</td>
</tr>
</tbody>
</table>

*Adapted from Reference 10.

### TABLE 8.9ddd

<table>
<thead>
<tr>
<th>Flow Transmitter</th>
<th>Flow Totalizer</th>
<th>Solenoid Valve</th>
<th>On/Off Valve</th>
</tr>
</thead>
<tbody>
<tr>
<td>Failures/10(^5)</td>
<td>10</td>
<td>10</td>
<td>30</td>
</tr>
<tr>
<td>Operations/Process Cycle</td>
<td>100</td>
<td>100</td>
<td>2</td>
</tr>
<tr>
<td>Mean Cycles between Failure</td>
<td>1,000</td>
<td>1,000</td>
<td>16,000</td>
</tr>
<tr>
<td>Mean Time between Failure, Days</td>
<td>250</td>
<td>250</td>
<td>4,000</td>
</tr>
</tbody>
</table>
8.9 Chemical Reactors: Basic Control Strategies


**Bibliography**