12

SUCCESSFUL PLANT TESTS

12.1 INTRODUCTION

Step 4 of the five-step problem-solving procedure often involves a plant test of some kind. These tests can vary from very straightforward to very complicated. While some of these comments were included in Chapter 3, conducting a successful plant test is so important to solving many problems that it deserves a separate chapter.

The reader of this chapter may or may not be involved in planning a plant test. However, it is likely that he will be involved in planning, reviewing, or executing of the test. Thus it is important that he understand the criteria and the elements involved in a successful plant test.

As indicated earlier, a successful plant test is one that either confirms or disproves the hypothesis. An unsuccessful plant test is one that fails to either confirm or negate the hypothesis. In addition, it is important that the plant test be conducted in such a fashion that it does not create a major problem for plant operations. The concept of analyzing problem solutions for potential problems was discussed earlier. Before conducting any plant test, a well-thought-out potential problem analysis should be completed.

The Russian nuclear power plant disaster at Chernobyl was at least partially due to failure to complete a well-thought-out potential problem analysis ahead of the test. The test was designed to determine whether, in the event of a

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reactor shutdown, enough power was available to operate the emergency equipment and core cooling pumps until the diesel power supply came online. It was to be conducted during the planned shutdown of reactor 4. The following contributed to the disaster:

- Only six to eight control rods were used during the test, despite there being a standard order stating that a minimum of 30 rods were required to maintain control.
- The reactor's emergency cooling system was disabled.
- The test was carried out without a proper exchange of information between the team in charge of the test and personnel responsible for the operation of the nuclear reactor.

12.2 INGREDIENTS FOR SUCCESSFUL PLANT TESTS

All successful plant tests will require the following:

- A full evaluation of instruments and laboratory procedures to be used for the test.
- A careful statement of what results are anticipated and how the anticipated results will be evaluated. This will almost always involve a significant amount of pretest calculations.
- A complete and well-thought-out potential problem analysis including "trigger points" which, if violated, will cause the test to be terminated.
- A careful and detailed explanation to operating personnel about the test.
- A formal post-test evaluation and documentation.

These items are discussed in more detail in the following paragraphs.

12.3 PRETEST INSTRUMENT AND LABORATORY PROCEDURE EVALUATION

One might think an evaluation of instrumentation prior to a plant test would involve only instrument technicians "zeroing" and calibrating instruments. In addition, however, the instrument specification sheets themselves should also be reviewed if there is any doubt at all about the instrument validity. For example, as discussed in Chapter 11, during a plant test in a high pressure process, it can be difficult to get a good material balance using instrumentation. All instrumentation was checked and confirmed to be as indicated on the specification sheets. On a closer evaluation of the specification sheets, it was discovered that no allowance had been made for gas compressibility at the high pressures. This would result in a higher density than that calculated using the ideal gas relationships. There will probably be value in reviewing the specification sheets of all new instruments to be used in a plant test. Depending on the complexity of the instrument specification sheets, this review may require the assistance of a process or instrument engineer.

While laboratory procedures may be thought of as the domain of the chemists, if results do not appear to make sense, the problem solver should not assume that the procedure is correct or is being correctly followed. As an example, during a startup of a chloride removal system, strange laboratory results were reported. The procedure developed to test for the level of the chloride ion called for acidification (reducing the pH into the acid range) of the sample prior to running the test for chlorides. The laboratory technician used the most available acid (hydrochloric acid) for the acidification step. This was discovered only when a tactful process engineer asked if he could watch the laboratory technician perform the test. On careful examination of the procedure, a footnote was found that indicated that nitric acid should be used in the acidification step.

These two examples are provided to indicate how the process engineer or problem solver must be involved in every step of the problem-solving efforts and/or plant test preparations, regardless of which discipline has primary responsibility. In these two cases, a more thorough review of procedures prior to the plant tests would have allowed the problem solver to discover that the incorrect gas compressibility was used as the basis for flow meter calculations and that the laboratory procedure was not as clear as it should have been. That is, the procedure calling for the use of nitric acid should not have been in a footnote.

12.4 STATEMENT OF ANTICIPATED RESULTS

Essentially, all plant tests are directed toward improving plant operations. However, in order to obtain an adequate evaluation of the plant test, it is imperative that the anticipated results be stated as quantitatively as possible. In addition, the variable or variables to be used to monitor the results of the plant test should be quantified. Both the positive and potential negative results should be spelled out. For example, a plant test on operating a fractionation column at a 15% increase in reflux rate to improve the purity of the distillate might have anticipated results stated as:

> "It is expected that this plant test will provide an increase in distillate purity of 0.5 weight percent. The distillate purity will be measured by the standard laboratory test procedure which is accurate to within 0.1 weight percent. An increase in tower pressure drop of 0.5 psi, with no indication of tower flooding, is anticipated. Tower flooding will be monitored by deviations from anticipated values of tray efficiency, tower pressure drop and heat balance closure."

There are several important aspects to notice in this statement. The fact that these are anticipated results means that calculations have been performed to determine the impact of the increase in reflux rate. For these calculations to be meaningful, similar calculations must be performed on the "base case." That is, fractionation calculations should be performed for the normal operating conditions. These fractionation calculations are based on theoretical trays. The base case tray efficiency can be determined by varying the number of theoretical trays in the calculations until the calculated compositions match the laboratory results. At this point, the tray efficiency can be determined by dividing the number of theoretical trays by the actual number of trays. This same tray efficiency can be utilized to estimate the change in distillate purity with the increased reflux rate.

If the anticipated distillate purity is not obtained, it is an indication that the tray efficiency has decreased. This could be the result of some kind of tray overloading condition. In a similar fashion, the tower heat balance closure can be determined for the base case. Heat balance closure is the difference between the heat added, including all heat sources (feeds and external heat source), and the heat removed with the products and cooling sources. In theory, this value should be zero. However, it is rarely zero, due to meter errors and heat losses. If the heat balance closure becomes worse as the reflux is increased, it is likely due to tower flooding. Tower flooding can cause liquid to be entrained from the top of the tower into the reflux accumulator. The heat balance calculations assume that anything that enters the reflux accumulator comes out of the tower as vapor. If some of the material going to the reflux accumulator is, in fact, liquid entrainment, the heat balance calculations will show more heat being removed from the condenser than is the actual case. This will result in a change in heat balance closure; this was illustrated in Chapter 8.

The fact that an anticipated tower pressure drop increase of 0.5 psi is indicated means that tray pressure drops have been calculated. The value in doing tray pressure drop calculations is twofold. It is necessary in order to assess whether tray flooding at the higher reflux rates would be anticipated. In addition, it provides an anticipated pressure drop which can serve as a trigger point to abort the test if exceeded.

While it is not anticipated that a process operator would have capability to perform these calculations, the calculations need to be done in order to set the basis for the plant test. These calculations can best be made by a graduate chemical engineer. The problem solver, whether he is an engineer or operator, needs to be aware of the need for these pretest calculations.

In addition to the pretest calculations, the anticipated results include a statement about the accuracy of the laboratory test. A plant test where the anticipated distillate purity increase is 0.5 weight percent would be meaningless if the laboratory accuracy was only ±0.5 weight percent.

The example of a fractionation column is very straightforward and easy to quantify with both statements and calculations. A more difficult plant test might be one in which a new catalyst with anticipated higher reactivity was to be tested. A similar technique to that described Chapter 9 could be utilized to determine the actual higher reactivity of the new catalyst. The simplified kinetic constant of the base case catalyst could be determined knowing the reactor residence time and the reactant concentrations. The simplified kinetic constant for the new catalyst could be determined using the same variables. A comparison of these two kinetic constants could be made to determine the increased reactivity of the new catalyst. While this is more involved than the less rigorous method of just comparing catalyst efficiencies (pounds of product produced per pound of catalyst) it avoids the need for the plant test to be run at the exact same residence time and reactant concentration. Plant tests of new, high-reactivity catalysts are often conducted at lower feed rate, production rate, and/or reactant concentration to avoid potential problems that might be associated with the higher reactivity. Catalyst efficiency will be impacted by these changes. Thus a comparison based only on catalyst efficiency will not be valid. This type of more complex plant test often involves considerations that, on the surface, seem difficult to assess. In many situations, it is possible to assess these considerations using fundamental chemical engineering skills. Two example problems are discussed later.

12.5 POTENTIAL PROBLEM ANALYSIS

A complete, well-thought-out potential problem analysis is mandatory. A trigger point should be developed for each major potential problem. If these trigger points are violated, the test will be terminated. Most of the variables considered in the statement of anticipated results will be considered in the potential problem analysis. While the concept of potential problem analysis is closely related to the previous discussion, there are some additional considerations. Some of these are related to safety. While the list below is not an all inclusive list, it is a list of the types of safety-related questions that should be considered:

1. Are any new chemicals being used?
2. Have byproduct reactions and byproducts been evaluated?
3. Are any new chemicals or reaction byproducts compatible with the existing materials of construction?
4. Are the test operating conditions outside acceptable ranges?
5. Are there any proposed conditions that will cause the safety release systems to be inadequate? For example, a higher reactivity catalyst might cause safety release facilities to be undersized.
6. Will operating condition changes cause a highly reactive chemical to concentrate to an unsafe level or be at unsafe conditions? Some examples are:
   - Oxygen concentrating in a vent to the point that the oxygen-hydrocarbon mixture is in the explosive range.

- Operating conditions for systems handling ethylene or acetylene reaching a pressure and/or temperature that can result in thermal decomposition.
- Operating temperatures for a thermally sensitive polymer being so high that decomposition, with release of highly reactive monomers, occurs.

7. Are there changes in operating conditions which seem innocuous, but could, along with a single unexpected occurrence such as a utility or mechanical failure, lead to catastrophic results?

The above list, as indicated, is not inclusive, but only serves as example of the types of safety items that should be considered. Any list of this type must not be considered a check list. It should be considered as a guide line only. Check lists often have a way of defeating their purpose by allowing the person responsible to simply check off items. In the pretest work for a plant test, serious consideration and significant calculations should be completed to ensure that no safety-related problems will occur.

In addition to safety-related items, an analysis should be made of what kind of things could go wrong (potential problems) during the plant test. As indicated earlier, each potential problem should have a trigger point. If this trigger point is violated, the plant test will be terminated. The value of having pretest trigger points is that they can be calculation- or logic-based when time is available for careful planning. This is opposed to waiting until unexpected events occur during the plant test and intuition becomes the mode of decision making. Examples of these trigger points are:

1. A new catalyst introduced into the reactor might lead to fouling of the heat transfer surface. In this example, the trigger point should be heat transfer coefficient. A trigger point of reactor temperature only will not be sufficient to determine if the heat transfer surface is fouling. The trigger point for the heat transfer coefficient should be set high enough to avoid the possibility that a small increase in catalyst rate will cause an uncontrolled increase in reactor temperature. Chapter 4 discussed a real-life example of this type of event.
2. A change in the reflux rate to a distillation column that should lead to an increased purity should include a trigger point to allow monitoring of unexpected tray performance deterioration. One possibility would be a trigger point specification on column pressure drop. The anticipated affect on column pressure drop could be calculated prior to the test and monitored during the test.
3. A plant test on adding a reagent to an exothermic batch reaction at a rapid rate might be monitored by a trigger point of temperature increase in the initial 2 or 3 min of reagent addition. Calculations prior to the test could determine the maximum temperature increase required to avoid

exceeding the maximum desired reactor temperature. If this calculated initial value was exceeded, the test would be terminated. Chapter 10 includes an example of this type of trigger point monitoring.

Hypothesis testing in a commercial plant may involve using results based on laboratory or pilot plant studies. Sometimes these results are based on equipment that does not simulate the commercial process exactly. A potential problem analysis should include possibilities associated with differences in equipment such as:

1. Did the reactor used in the studies simulate the commercial facilities? Considerations must be given to:
   - Simulation of a continuous reactor with a batch reactor.
   - Differences in mixing patterns and regimes in a laboratory or pilot plant reactor and a commercial reactor.
   - The reduced heat removal capability in the commercial reactor caused by the smaller heat transfer area to reactor volume (A/V ratio). Scaleup from pilot plant facilities to commercial facilities almost always involves a reduction in the A/V ratio, making heat removal more difficult.
2. Did the equipment used in the laboratory test adequately simulate commercial equipment? For example, chemical compound stability studies are often determined in a laboratory oven. If the material will be subject to high shear rates in the commercial equipment, laboratory oven studies may not be sufficient.

12.6 PLANT SPECIFIC REQUIREMENTS

The series of questions and comments listed above is not meant to supplant any specific company or operating plant requirements. Such steps as safe operating committee reviews, management of change reviews, OSHA-mandated reviews and/or peer reviews will still be required.

12.7 EXPLANATION TO OPERATING PERSONNEL

An explanation of the plant test should be given to operating personnel (and perhaps mechanical personnel). This explanation can be conducted in a training session, with a written handout, or through one-on-one discussions. The following items should be included:

1. *Purpose of the test*: The value of the test for the overall company objectives should be explained to the operating and mechanical personnel. The

plant test could be directed at solving an operating problem that will make for an easier job, or producing a superior product that will lead to increased sales. Regardless of the goal of the test, this is an opportunity to build enthusiasm for the test by explaining that the success of the enterprise leads to both job security and promotional growth for the individual.

2. *How safety was evaluated*: Unfortunately, most hourly personnel have had experiences with plant tests or plant changes where safety was not adequately considered. This explanation should consider new chemicals, new laboratory procedures, and new operating or mechanical conditions/procedures. Many operating companies have safety and peer review committees that review new operating changes. The operations and mechanical workers should be informed of the details of these reviews. In addition, if new chemicals are involved, the MSDS sheets should be reviewed with the appropriate personnel and posted in visible places.
3. *Why the test will work*: This will be an opportunity to explain to the hourly worker the theory behind the proposed test. Most theoretical explanations can be expressed in terms that even people without engineering degrees can understand. This is an important step and should not be considered just a requirement, but should be looked on as an opportunity to educate and obtain "buy-in" for the test.
4. *How the test will be evaluated*: The purpose of this explanation is twofold. It is an opportunity to explain exactly what will be considered in evaluating the test. It may be desirable, as part of the test, to have the hourly worker fill out data sheets. The accuracy of this will be a function of how well he understands how the values will be used. In addition, this will be a time to explain that a test that does not prove the hypothesis is not a failed test. The only failed tests are those that are not conclusive.
5. *If the test works, what he gets from it*: The hourly worker will often have questions such as:
   - If the test is successful and changes in operating or mechanical procedures are required, will it make my job easier or harder?
   - Will these changes reduce manning or limit future addition of jobs?
   - If the test is successful, how do I get any credit for doing all of this extra work?

These are questions that are often not brought up. However, they are almost always in the minds of the hourly workers. Ignoring them will usually leave it to the operator or mechanic to assume the worst case answers.

12.8 FORMAL POST TEST EVALUATION AND DOCUMENTATION

The formal post-test evaluation and documentation phase of the plant test is often one of the most overlooked areas of plant test execution. The desire to improve organization efficiency often creates pressures to move on to the next plant test involved with the current problem or to move on to the next problem to be solved. Whether the test proved or disproved the working hypothesis, the benefits of the documentation are to provide a lasting reference that will help avoid repeating the test or, even worse, avoid future changes that will cause the problem to recur. In addition, as indicated earlier, the writing process will often clarify the thinking process and improve problem-solving activity.

A plant test that disproves the hypothesis is often not documented because the problem solver does not see this as important. He feels the need to move on and work on the next hypothesis to get to a problem solution. The failure to document successful plant tests that disprove the hypothesis will often cause a similar hypothesis to be proposed at some future time. Thus the failure to document the work, because of the alleged need to be efficient, will cause a loss in organization efficiency. A comment often made by many experienced operating and/or mechanical personnel when presented with a proposed plant test is, "We tried that before and it didn't work." This is an indication that formal documentation of previous tests is not being done.

Even more serious is the failure to document a plant test that proved a working hypothesis. In this scenario, changes are made which eliminate the problem. Several years later, a proposal is made to reverse the changes to try to solve another problem, increase production, or improve product quality. When the question "Why are we operating at these particular conditions?" is raised, no documentation exists to show the fact that changes were made to eliminate an operating problem. Thus the assumption is made that the conditions can be returned to the previous conditions.

Another scenario is the case of a failed plant test. That is one that neither proves or disproves the working hypothesis. Rather than making another attempt with an improved plant test that will be successful (prove or disprove the hypothesis), the approach is just completely abandoned. No documentation is done to indicate why the test was not conclusive. At a later point in time, the hypothesis is reintroduced. When a similar test is proposed, the memories of both technical and operating personnel are that "We tried it before and it didn't work." Actually, the validity of the hypothesis is unknown. What is known is that the test did not prove that the hypothesis was correct. The converse, that the test proved that the hypothesis was incorrect, is not true. Because the recollection is that the test did not work, the hypothesis is abandoned. A good documentation of the previous test would have indicated that what failed was the test itself, rather than the correctness of the hypothesis.

The size of the actual document should be minimized. It should include items such as the objective of the test, the test procedure, the test results, and the conclusions. In addition, any comments concerning safety should be included. Because of the technical conclusions and the possible need to incorporate changes into operating conditions or procedures, the final document should be approved by both operating and technical management.

12.9 EXAMPLES OF PLANT TESTS

Some plant tests to verify hypotheses will be simple and some will be more complicated. The next few pages present two examples of involved plant tests that were directed at improving a product and/or process. While they are not directly related to solving plant problems, they are real world examples that illustrate the concepts discussed above. These same concepts are applicable to plant tests directly associated with solving plant operating problems.

EXAMPLE PLANT TEST 12-1

A synthetic rubber producer was pressured by customers to change their stabilizer to one that would continue providing a product that did not discolor due to stabilizer oxidation, but would have better product-related properties. Based on the customer request, a new stabilizer was identified by the technology organization. Tests to confirm the stabilizing and nondiscoloring properties were conducted at different temperatures in laboratory ovens. These tests were conducted by first mixing the stabilizer with the polymer at ambient temperatures in low-intensity mixers. The polymer containing the stabilizer was then heated and held at elevated temperatures in the oven. These tests confirmed that the product did not discolor and that the molecular weight of the polymer did not change even after several hours in an oven. Thus the new stabilizer seemed to meet the goals, that is, the product did not discolor and the molecular weight did not decrease, indicating that the new additive was an effective stabilizer.

The actual synthetic rubber process consisted of a polymerization section where the stabilizer was added to a slurry of rubber and water. This slurry was then pumped to the finishing section where the rubber was dried to remove water, extruded, and rolled on hot mills to put the final product into a form that could be boxed and shipped to the customer.

A plant test was scheduled to assess the utilization of this new stabilizer in the plant. The hypothesis being tested was that the new stabilizer could be added effectively in the plant and that it would be as efficient as indicated in the laboratory results. Since the laboratory data indicated that the stabilizer was very effective, only some of the steps listed above were implemented. These steps and what was actually done are summarized below:

1. *Pretest Instrument and Laboratory Procedure Evaluation*: For the most part, this was done in a satisfactory manner. The laboratory was prepared to analyze the stabilizer and, in addition, instruments were adjusted for the slightly different density.
2. *Statement of Anticipated Results*: Since the technology experiments indicated that such good results were obtained, very little was done to

prepare a statement of anticipated results. It was just assumed that the stabilization properties would be comparable to those of the existing stabilizer, or even better. That is, that there would be no change in molecular weight during the finishing operation.

3. *Potential Problem Analysis*: Actually, very little was done to develop a potential problem analysis. The technology work was so convincing that this new stabilizer would meet the criteria of not discoloring and avoiding molecular weight change in the finishing operation that this phase was just ignored. It was anticipated to be a "boring test."
4. *Explanation to Operating Personnel*: This was not done.
5. *Formal Post-test Evaluation and Documentation*: As discussed below, the test disproved the hypothesis so conclusively that it was mandatory to document the results of the test.

Since, based on technology testing, it appeared that the new stabilizer had stabilizing properties equivalent to those of the current stabilizer, all operating conditions such as polymer molecular weight and amount of stabilizer added were held constant and operations were simply switched over to the new material. Very soon, the molecular weight of the product leaving the finishing operations began to decrease rapidly, even though the molecular weight in the polymerization operations did not change. This was completely the opposite of what would be expected if the stabilizer was performing as anticipated based on the laboratory tests. The lack of a well-thought-out potential problem analysis before the test was now causing a panicked problem-solving attempt during the test. The two different groups involved (operations and technical) began pursuing different approaches.

The operating group increased the molecular weight in the polymerization section in an attempt to make on-specification product leaving the finishing section. Since the molecular weight in the polymerization process was inversely proportional to the monomer conversion, it was necessary to reduce the monomer conversion in order to increase the molecular weight. This lowered monomer conversion tended to overload the monomer recycle system. The operations continued to deteriorate. The molecular weight of the finished product continued to fall and the monomer conversion continued to be decreased as a compensatory action. The test was aborted after only a few hours of operation.

The technical/technology group initiated problem-solving activities. An analysis of the final product indicated that there was very little stabilizer present. This brought up several potential hypotheses:

- An incorrect amount of stabilizer was being added.
- The stabilizer was being washed off the polymer in the water slurry operation.
- The stabilizer was being vaporized in the finishing operation, even though long-term technology tests in the ovens indicated this would not occur.

- The laboratory results were wrong and stabilizer really was present at the desired concentration.
- There was the target amount of stabilizer in the polymer; it was just not an effective stabilizer. This would be in conflict with the technology data.

An evaluation of the data after the test was aborted indicated that the stabilizer was being added at a correct rate and was not being washed off the polymer in the water slurry operation. However, it was being vaporized in the high shear zone of the extruder and hot mills. These zones were not only at elevated temperatures, but the high shear caused large amounts of the surface to be exposed to the atmosphere. This was a radically different condition than what was experienced in the low-shear environment of the ovens.

Lessons Learned There are several lessons that can be learned from this plant test. They involve areas such as the failure to conduct a potential problem analysis, the failure to provide an explanation to operating personnel, the blind acceptance of technology data without questioning the basis for conclusions, and the attitude that this is going to be a "boring plant test."

A potential problem analysis would have very likely uncovered that the shear rates in the extruder/mills were vastly different than those in the very low-shear drying ovens. This consideration would have generated additional technology work to try to simulate the high shear rate. This additional work would have likely shown that large amounts of stabilizer were lost in the high-shear-rate studies. If the proposed stabilizer still appeared to be the best available option, the plant test could have been conducted at much higher stabilizer addition rates to compensate for the vaporization of the stabilizer in the extruders and mills. A potential problem analysis would have also led to consideration of the overloading of the monomer recycle system that occurred when the conversion had to be dropped to a low level to obtain the specification molecular weight in the finished product. Plant tests where technology data is accepted blindly and/or where the test is considered to be boring will almost always result in failure of some kind.

The lack of a careful and detailed explanation of the plant test to the operating personnel did not impact the results of this first test. However, when another test was scheduled, there was a considerable amount of tension caused by the failure to adequately explain the goals and purposes of the first test.

EXAMPLE PLANT TEST 12-2

As a second illustration of a plant test, consider the following example. It was desirable to test the capacity of a single reactor in a plant that used two reactors in series. This would allow assessment of the possible, low-cost debottlenecking of the existing plant as opposed to the alternative of building a new plant. If the single reactor could be operated at the same capacity as the exist-

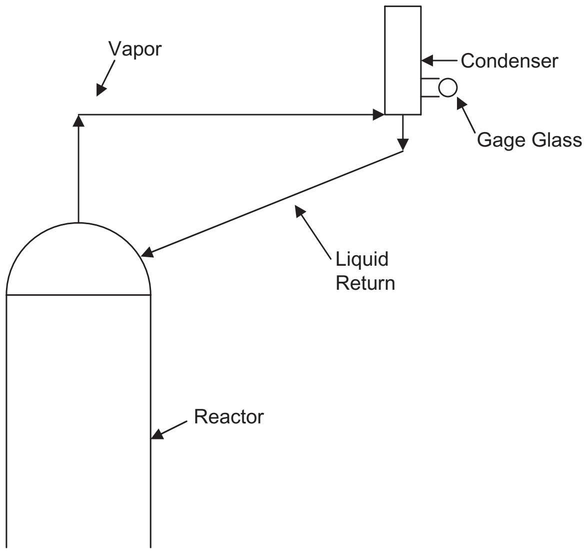

Figure 12-1 Reactor overhead condenser schematic.

ing two reactors in series, it would be possible to increase the existing plant capacity by at least 50%. The hypothesis to be tested was that a single reactor could produce at the same production rate as two reactors in series with no increase in fouling rate.

The reactor was a boiling reactor where the heat of reaction was removed by the vaporization of the reactant, which was then condensed and returned to the reactor. The reaction product consisted of small, insoluble, solid particles. Because of the size of the particles, some were always entrained with the vapors leaving the reactor. The vertical condenser used to condense the reactant was provided with trays to allow the entrained solids to be washed back into the reactor. A sketch of this system is shown in Figure 12-1.

A plant test was deemed necessary since, at the higher vapor rates associated with the higher reactor capacity, more entrainment might be encountered. The increased entrainment might lead to fouling of the exchanger, the vapor line, or the liquid return line. In addition, the liquid handling capacity of the return line under possible fouling conditions was uncertain. It was necessary to develop techniques for monitoring the possible fouling of the exchanger, as well as the vapor line and liquid return line.

Three techniques were developed to monitor operations and fouling during the test of the single reactor. They were as follows:

1. The heat transfer coefficient on the overhead condenser was monitored hourly by the process control computer.
2. A local level gauge on the bottom of the exchanger was monitored every 2 hours by visual observations. An increase in the liquid level in the

bottom of the exchanger would indicate that the condensate was not flowing back into the reactor freely. That would mean that the liquid return line was likely partially plugged.

3. The vapor and liquid lines to and from the exchanger were "rung" every 2 hours. This "ringing" was a process of lightly tapping on the line with a hammer. If fouling was beginning to occur in the line, the pitch of the ring would have changed from a sharp, clear ring to a dull thud. While this qualitative test was not described in text books, it had been proven by operating personnel in earlier experiences.

The manual monitoring (local level gauge and "ringing the lines") required the operating personnel to climb six flights of stairs in order to reach the desired physical area of the equipment.

As indicated earlier, there are five aspects essential to a successful plant test. For this plant test, these items were covered as follows:

1. *Pretest Instrument and Laboratory Procedure Evaluation*: This was relatively simple for this test. The calibration of instrumentation that would allow personnel to determine the exchanger heat transfer coefficient was part of a routine task order. However, as indicated below, it was necessary to re-range several instruments to allow operation of the reactor at higher capacity. The manual procedures for checking the level and condition of the vapor and liquid lines were well established by previous experiences with solids entrainment.
2. *Statement of Anticipated Results*: Prior to the plant test, the heat transfer coefficient on the condenser was determined from the existing temperatures and heat duty. Calculations were made to determine what the anticipated inlet and outlet water temperatures would be at the anticipated heat duty. These calculations assumed that the heat transfer coefficient would be slightly lower than that determined at the existing duty. This was done to allow for a higher thickness of condensate flowing down the tubes. The actual change in condensate thickness would be very difficult to calculate with any accuracy. Thus the reduction in overall heat transfer coefficient caused by the thicker condensate layer was assumed to be 10%. Calculations were also made to confirm that the vapor and liquid lines had adequate capacity in their clean conditions. Operations personnel had an intuitive feel for what a clean line sounded like when it was rung. Thus an overall statement of anticipated results would indicate the following evaluation of the reactor overhead circuit was expected:
   - Based on the anticipated heat transfer coefficient, the anticipated values of the water temperatures and flow rates around the condenser were estimated. These were included in the anticipated results.
   - Based on calculations, there was anticipated to be no level in the bottom of the exchanger. In this case, the calculations indicated that

the liquid and vapor lines in a clean condition had sufficient capacity, so that there should be no level buildup in the exchanger.

- The overall statement included a comment that the vapor and liquid return lines should ring with a clear sound.

As with any process equipment or operating condition changes, safety must be considered. At the higher reaction rates, the safety valves would be required to release at a higher rate in an emergency. The required and actual capacities of the valves were reviewed and it was found that the capacity of the valves was greater than the maximum anticipated release rate. After other potential safety problems had been evaluated, a statement was provided indicating that no safety problems were anticipated.

Other areas of the process were also considered to evaluate the effect of operating at full rates with only a single reactor. An evaluation of the catalyst feed system to ensure that adequate catalyst supply would be available was conducted. The catalyst efficiency was anticipated to decrease as reactor volume was removed from service. All of the instrumentation required for the reactor to be operated at the higher rate was evaluated and meter ranges were increased where necessary.

3. *Potential Problem Analysis*: In the case of this plant test, there were potential problems associated with the overhead condenser as well as at other locations in the plant. Trigger points were developed for the heat transfer coefficient on the overhead condenser. This variable was routinely calculated by the process control computer and a trigger point was set to ensure that the reactor temperature did not run away. A reactor temperature runaway could occur if the heat transfer coefficient on the condenser is so low that a slight increase in reaction rate would cause the heat generated to increase at a faster rate than heat removal capabilities were able to handle. This was explained in detail in Chapters 4 and 10. The trigger points for the liquid level in the condenser and for the ringing of the lines were more qualitative. Any accumulation of liquid in the condenser or line ringing that was not clear were set as trigger points.

When operators focus on certain areas of the plant for a plant test, other areas are often overlooked. As indicated above, the catalyst feed rate was anticipated to increase due to the reduced residence time associated with using a single reactor. Since the reactor was a boiling reactor, some of the reactor volume below the liquid level was bubbling vapor. As such it would not be an effective reaction volume. Correlations were used to estimate the effect of the higher "boil up" rate associated with the increase in production on the bubbling vapor volume of the reactor. As the boil up rate increased, the correlations predicted that the bubbling vapor volume below the liquid level would increase and the effective reaction volume would decrease. The potential problem that these correlations were inaccurate was considered. If the reduction in reactor volume associated with the bubbling vapor was greater than expected, the catalyst efficiency would be lower than anticipated. Thus a trigger point was included for catalyst efficiency. Trigger points were determined for other parts of the process where potential problems were anticipated.

The key idea to consider is that having the well-thought-out potential problem analysis encourages reacting to situations in a predetermined way rather than in a reactionary mode in the midst of a problem.

4. *Explanation to Operating Personnel*: Most operating and mechanical personnel were happy to hear that their efforts to make a good product resulted in consideration of an increase in capacity. However, they had concerns that had to be addressed in order to obtain their cooperation for the plant test. Some of these comments and concerns were:
   - "We don't have time to climb stairs and do the checks every two hours."
   - "You guys will get all the credit while we do all the work."
   - "Won't running the plant at a higher rate make more work for me?"
   - "If we expand by adding more plants that will mean more jobs for my friends and family. This 'debottlenecking' expansion doesn't add any jobs."
   - "Is it really safe to operate the reactor in this fashion?"
   - "I don't see how we can run this reactor at higher rates when we are having problems with carryover at the lower rates."

   Essentially, all of the questions that might come from the operating or mechanical personnel fall into two categories. There are questions that can be answered based on calculations that have already been done as part of the pretest efforts or potential problem analysis. The value of doing the calculations is that the operator can be told, "We have considered that and it is not a potential problem." The question of reactor safety is an example of this. It is always possible that a question will be raised which has not been considered. Serious consideration should be given to any question of this nature.

   There will also be questions that cannot be answered based on calculations. Many of these will require an explanation that deals with company goals or the competitive situation. For example, the desire to expand by building more small plants could be answered by the fact that competitors are building larger plants and that being competitive with them provides job security. Job security for the employee is more important than creating jobs for friends and family.

5. *Formal Post-test Evaluation and Documentation*: While the test appeared to confirm the hypothesis, a formal test document was still prepared.

> The formal test evaluation and documentation included the conclusion that the heat transfer coefficient of the condenser at the higher rates was comparable to that at the normal rates. The slightly higher condensate thickness did not cause any decrease in heat transfer coefficient. When the heat transfer coefficient was evaluated as a function of time, there was no indication that fouling was occurring during the test. In addition, there was no indication of fouling as gauged by the absence of any level in the condenser and the clear sound of the pipe ringing during the test.

The catalyst efficiency anticipated at the higher rates was compared to the actual catalyst efficiency. Since these values were very close, it was concluded that no unanticipated loss in reactor volume associated with vapor bubbling occurred during the test.

Perhaps one aspect that is often overlooked in documenting a plant test is the comments from mechanics or operating personnel. Comments of operating personnel were included. Their comments were that things ran very smoothly during the test.

Lessons Learned This test was successful from the standpoint that the results indicated conclusively that it would be possible to operate the reactors at significantly higher rates. Even if the conclusions had been that it would not be possible to operate the reactors at high rates, the test would still have been successful; the results would have been conclusive. As indicated earlier, a successful plant test is one where the test result is conclusive.

Several lessons were learned from this test. While the time to do the pretest calculations and potential problem analysis may seem to be inefficient, it provided a basis to convince management and operations personnel of the feasibility of the test. It also provided a basis for determining trigger points to allow a predetermined decision on aborting the test.

Using any short-term test to make decisions about fouling is difficult. The risk of concluding that fouling was not occurring was ameliorated by obtaining the maximum amount of data. In this case, three different techniques were used to monitor fouling.

There are always potential problems associated with any plant test. In the earlier example (12-1), the anticipation that the test was going to be boring was radically different than the anticipation for this test.

Even in tests that appear to confirm a hypothesis, documentation of the results is important. In this test, it was obvious that the results associated with fouling should be documented. In addition, the analysis of the expected catalyst efficiency at the higher rates and the higher boilup rate proved very valuable. Without this analysis and documentation, erroneous conclusions may have been reached regarding the reduced catalyst efficiency encountered as rates were increased.

While the discussion of pretest calculations indicated several technical procedures that a process operator might not have the training to perform, they are included here to illustrate what kind of calculations should be made prior to a plant test. Whether or not the process operator serving as a problem solver has the skills to make certain calculations, he should insist that the calculations be done prior to a plant test. An example of this in Example 12-2 is the calculation of the safety valve capacity as well as the safety valve release rates at the higher reactor production rates.

12.10 MORE COMPLICATED PLANT TESTS

It will frequently be necessary to consider conducting a plant test where the cost of a plant shutdown is so great that in-depth theoretical comparative studies must be done as part of the potential problem analysis. These studies will allow determining the risk of such an event by a comparison to existing operations. These comparative studies will not provide absolute values, but will allow determination of whether the plant test will increase or decrease the item of concern. An example of this might be a gas phase reaction process in which the presence of fines (small particles) could cause entrainment with a subsequent plant shutdown. Fines are known to be present with the current catalyst. However, the exact concentration is not known. The level of fines associated with the current catalyst results in minimal shutdowns. There are theoretical techniques available to predict the amount of fines produced by the catalyst, knowing the catalyst attributes (reactivity, particle size, and particle size distribution) and reaction conditions.

A plant test using a new catalyst with radically different attributes in the gas phase reactor was proposed. Concerns were expressed about the potential for this test to cause a plant shutdown. In this case, the absolute level of fines was not as important as answering the question of "Will the amount of fines increase or decrease?" In order to assess the risk of a plant shutdown if a plant test is run with the new catalyst, calculations were done to estimate the amount of fines produced by both the existing and new catalysts. These calculations were then compared and an assessment made regarding the possibility of increased fines. If this assessment indicated that there will be a comparable or lower level of fines produced, then there is a high probability that the plant test can be conducted without shutting down the plant. Conversely, if the calculations indicate that the fines level will increase, a plant shutdown is a definite concern. In this case, it might be better to redesign the catalyst rather than risk a plant shutdown caused by the test of a new catalyst.

12.11 DESIGN OF EXPERIMENTS (DOE)

During a laboratory investigation of a new process, new catalyst, or new product, it is often desirable to conduct a series of carefully planned experiments. This approach is referred to as a design of experiments (DOE). When one is working to solve a plant problem, the DOE approach is often unnecessary and is often counterproductive. There are three reasons for this:

- The range of variables in an operating plant is normally very broad. Thus data is available over almost any range that would be covered by a proposed DOE.
- DOEs are most applicable when there is no proposed hypothesis. Experiments must be conducted before one can even begin to develop a hypothesis. When one is solving a plant problem, the potential hypothesis is relatively easy to develop. But it must be proven by calculations and/or a plant test.
- A true DOE will often cause a plant to operate at conditions that are outside of the stability limit, resulting in plant upsets or shutdowns.

12.12 KEY PLANT TEST CONSIDERATIONS

The key idea to remember from this chapter is that if a plant test is used to confirm the hypothesis, it must be well planned and aim to thoroughly prove or disprove the hypothesis. Successful plant tests are those that either confirm or reject the proposed hypothesis. Documentation of the plant test results, regardless of the conclusion, is also imperative.