Using Design of Experiments to Capture Valid Data from Pilot Plants

By Ron Stites
Managing Member
Stites & Associates, LLC
Brighton, Colorado
www.tek-dev.net

Pilot plants are generally defined as plants that are bigger than lab-scale, yet smaller than commercial demonstration or semi-works plants. They are primarily designed to collect data rather than to make product. The pilot plant can often collect information that cannot be captured at a smaller scale. For example, heat and energy balances are difficult to scale, especially for high exo- or endothermic processes. Commercial plants are so expensive to build that they rarely are built without convincing pilot plant data. A commercial oil shale operation requiring hundreds of millions of dollars of investment must have valid pilot plant data to justify that investment.

One reason for running a pilot plant is to make sure that the process works and is safe. But the fact that a process works doesn't necessarily mean that it can make money. Measurements that can accurately predict the financial performance of the plant are critical. The potential exists for pilot plant data to be used to reinforce opinions, prejudices and hopes, so it's important to confirm the statistical validity of the results. Furthermore, pilot plants are expensive to operate so the number of experimental runs must be kept to a minimum.

Planned experiments are crucial to gathering meaningful data from pilot plant operations. Variables need to be identified in advance and controlled in order to gather objective data. The information gained from intuitive action, including general shakedown and getting a feel for things, is useful but should not be considered learning. Backward looking testing can only develop plausible hypotheses to be tested in the future. Changing one factor at a time is rarely effective because it requires a large number of runs and fails to reveal factor interactions. On the other hand, managing multiple factors requires discipline. Thinking about interactions between factors encourages thorough upfront research and consideration of actual causality which is frequently nonlinear. The gold standard is design of experiments (DOE), a process in which you define factors to manipulate and responses to measure and then use statistical analysis to define the relationship between the results and factors and determine whether or not the results are statistically significant.

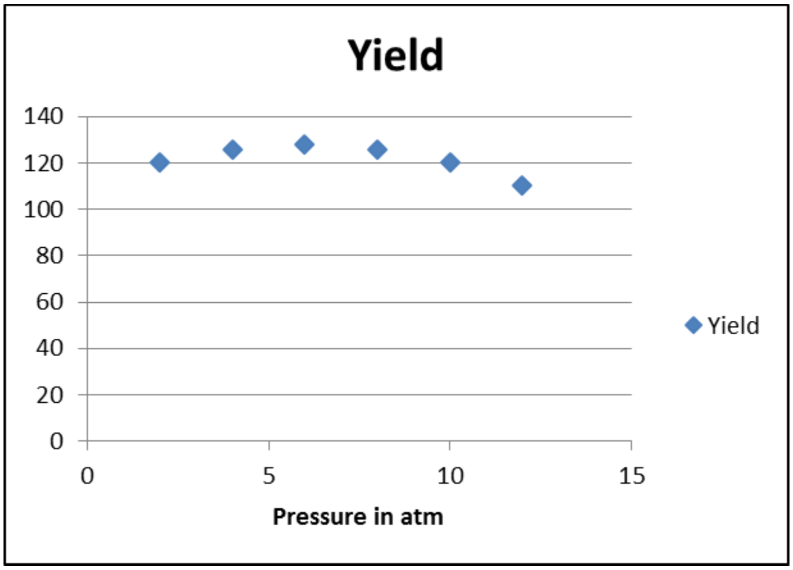

Figure 1: Pressure versus yield curve obtained by running experiments starting with the lowest pressure and progressively increasing pressure

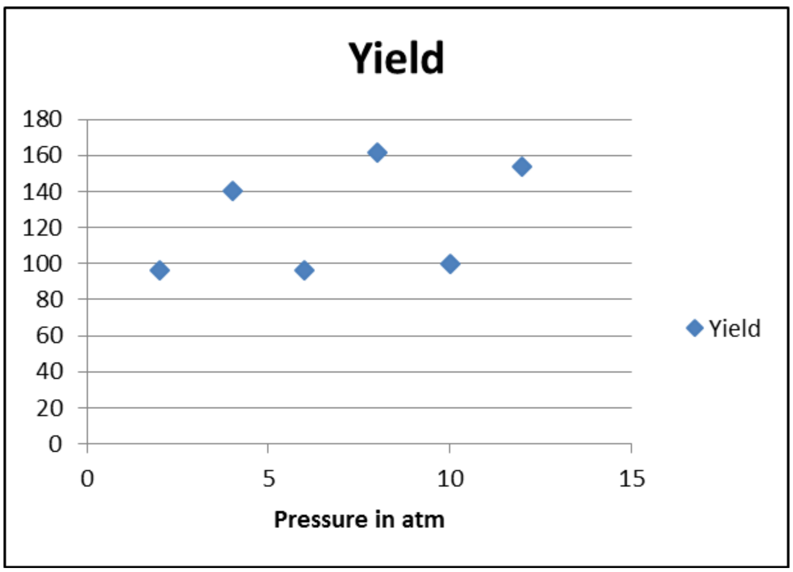

Figure 2: Pressure versus yield curve obtained by randomizing the order of experiments

Whenever possible the manipulation of factors should be randomized. The example shown in Figures 1 and 2 demonstrates why this is important. Let's suppose that we are testing a catalyst for making product at various pressures. In this case it is a lot easier when running experiments to start at low pressure and then go to high pressure. Ordering the experiments this way provides the smooth curve shown in Figure 1 and leads us to conclude that there is a maximum around 6 atm. What we didn't realize was that the activity of the catalyst was going down with time. Running the test in random order by pressure yields the graph shown in Figure 2. This graph indicates clearly that one or more factors besides pressure are playing an important role.

The rest of this article will present several real examples of how DOE was used in pilot plants to quantify the effects of the factors, singly and in combination, while ensuring the statistical validity of the results. These examples all used Design-Expert® software from Stat-Ease, Inc., Minneapolis, Minnesota, www.statease.com. This DOE software is designed for use by subject matter experts who are not necessarily experts in statistical methods. The software walks users through the process of designing and running the experiment and evaluating the results. Stat-Ease also provides very good support. I contact them not only for questions about using the software, but also to check out my statistical thinking and they have always been very helpful.

The first example involves a pilot plant designed to study carbon sequestration using an organic compound to absorb CO2 from flue gas. During early runs, a white precipitate formed under certain conditions. Engineers used DOE to manage a series of tests for the purpose of understanding under what conditions the precipitate would form. The experiment showed that the precipitate could be eliminated by controlling the amount of water used in the reaction. Furthermore, optimizing the water content not only eliminated the precipitate but increased the capability of the process to capture CO2. The result was a significant increase in the performance/cost ratio of the new process.

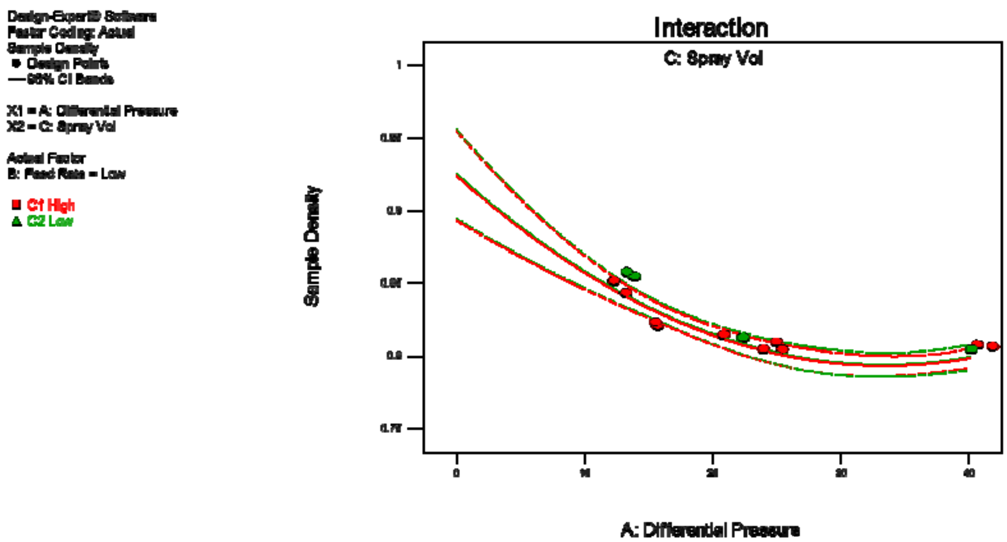

Figure 3: DOE shows a nonlinear relationship between pressure and sample density in oil mist recovery system

The second example involves the application of DOE in a pilot plant to optimize an oil mist recovery system that was set up in an effort to improve the efficiency of a large power plant. A literature search revealed that product collection was most likely dependent upon velocities and pressure drops and was nonlinear in its response. But increasing the pressure increases the amount of energy required to operate the recovery system which in turn increases operating costs. DOE was used to model the sample density—the inverse of product recovery—as a function of the differential pressure and the spray volume. As shown in Figure 3, the results identified interactions between the factors that would have made it impossible to optimize their values by testing each variable in isolation. With DOE, engineers were able to find the lowest possible pressure drop that provided a suitably high yield while running the minimum number of tests.

In a third case, another pilot plant was put together to evaluate a new catalyst in a process that involves converting syngas to alcohol that could be used as fuel. Many factors were considered including the H2/CO ratio, temperature, gas hourly space velocity—a measure of how fast gas flows over the catalyst, and the proportion of gas that was recycled. A typical DOE is shown in Figure 4. The primary response was the alcohol yield but other responses included the product composition—the proportion of methanol vs. ethanol, tail gas composition and the rate at which the catalyst degrades. DOE optimized the performance of the catalyst and led to the development of a new method to prevent catalyst degradation. The catalyst itself turned out not to be viable, however, the patent on the method to stop deactivation of the catalyst was sold for several million dollars.

Std	Run	Factor 1 A:H2/CO Ratio mole ratio	Factor 2 B:GHSV V/V ratio	Factor 3 C:Temperature deg C	Response 1 MeOH/EtOH mole ratio	Response 2 Yield gEtOH/kg-hr
6	1	3:1	10000	300		
3	2	2:1	5000	300		
15	3	2:1	5000	340		
4	4	3:1	5000	300		
14	5	3:1	2000	340		
2	6	3:1	2000	300		
18	7	3:1	10000	340		
10	8	3:1	5000	320		
1	9	2:1	2000	300		
16	10	3:1	5000	340		
11	11	2:1	10000	320		
7	12	2:1	2000	320		
12	13	3:1	10000	320		
5	14	2:1	10000	300		
17	15	2:1	10000	340		
9	16	2:1	5000	320		
8	17	3:1	2000	320		
13	18	2:1	2000	340		

Figure 4: A typical run plan for a catalyst test

In a fourth example, a company developing a device to improve ethanol yield performed trials without using DOE at several operating plants that showed promising results. The company brought the device to market and sold several large systems. Unfortunately, the results did not measure up to the trials. At this point, the company re-ran the trials using DOE and, as shown in Figure 5, discovered that the original positive results could not be reproduced and were most likely just statistical noise. The designed experiment showed that the impact of the new device on the process was actually negative. The company abandoned the market at considerable expense but DOE saved money by avoiding additional investment that would otherwise have been made in further efforts to reproduce the original positive results. The application demonstrates that poorly designed and executed trials can have very negative consequences.

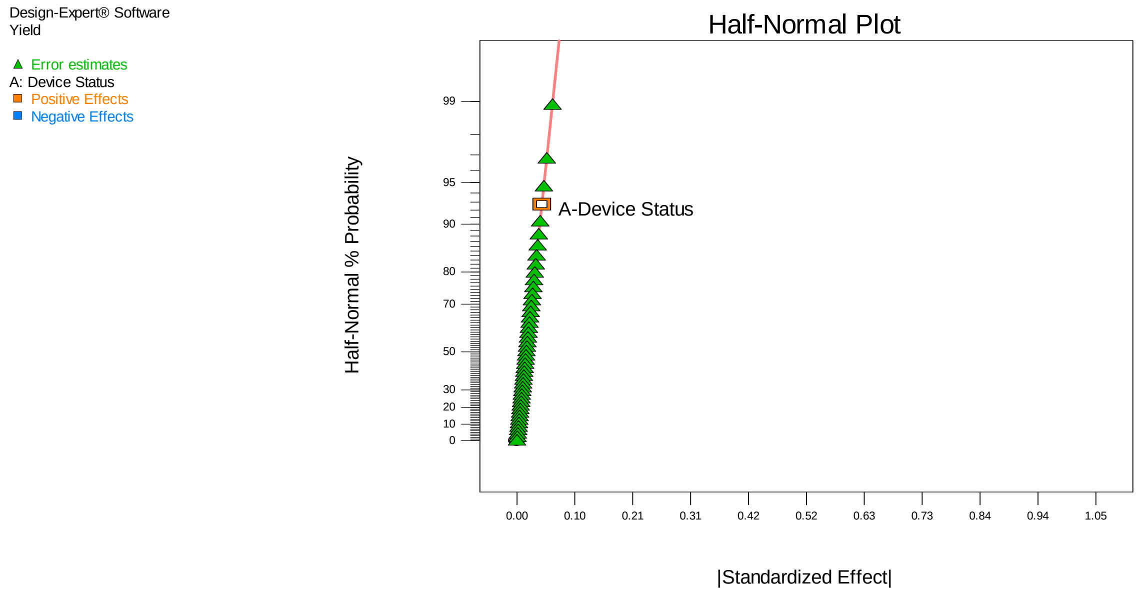

Figure 5: A half-normal plot showing that the device effects were approximately random

Pilot plants are expensive to operate so it's important to gain understanding from their operation in the minimum amount of time. On the other hand, commercial plants are even more expensive to build so ensuring that lessons learned from the pilot plant are valid and reproducible is absolutely essential. DOE can help pilot plant operators address both of these challenges. A designed experiment minimizes the number of runs required to establish the relationship between the factors and the responses. At the same time, DOE provides the rigor that establishes benefit/risk relationships with statistical validity to provide the precision and accuracy that should be the basic criteria for commercial plant investment.

Stites & Associates, LLC contact information
Ron Stites, Managing Member, Stites & Associates, LLC; Brighton, CO. Cell: (912) 247-6120, Web Site: www.tek-dev.net, LinkedIn site: http://www.linkedin.com/pub/ron-stites/18/348/44a,
Blog: http://tek-dev.typepad.com/technology-development/

Stat-Ease contact information
Stat-Ease, Inc., 2021 E. Hennepin Ave., Suite 480, Minneapolis, MN 55413, USA, Web Site: www.statease.com, E-mail: info@statease.com