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Don't Be Fazed By Multiphase Sampling

An isokinetic system can provide accurate samples

By Andrew Sloley, Contributing Editor

OPERATIONS, TROUBLESHOOTING and even design may require sampling a multiphase stream — i.e., a solid in gas, solid in liquid, liquid in gas or liquid in liquid. Taking multiphase samples presents many challenges. The most difficult one is how to get a representative sample from the process. Often, the only apparent solution is to separate the two phases, measure each individually and then recombine them. However, this may be tough and expensive. Isokinetic sampling offers a possible way to take a single sample of a mixed-phase stream. Its typical current applications include dust in flue gas and entrained water in steam generators. The approach also has proven useful for other less-common problems such as entrainment in compressor feed drums, sand-in-oil entrainment and in feeds to oil/water separators and coalescers.

An isokinetic system takes samples with the same local velocity as the bulk flow at the sample point, as depicted in Figure 1 (a). This minimizes momentum effects between the phases. So, the sample contains the same proportion of both phases as the bulk flow. In contrast, if the sample velocity is lower than that of the bulk stream (b), the momentum of the higher density particles tends to keep them in the path to the sample nozzle, making the sample rich in particles. Conversely, if the sample velocity is much higher than that of the bulk stream (c), the momentum of the particles prevents them from going to the sample point, resulting in a sample rich in bulk phase.

Special probes that allow for isokinetic sampling commonly are used in both environmental applications and steam systems. For steam systems, ASTM D1066, "Standard Practice for

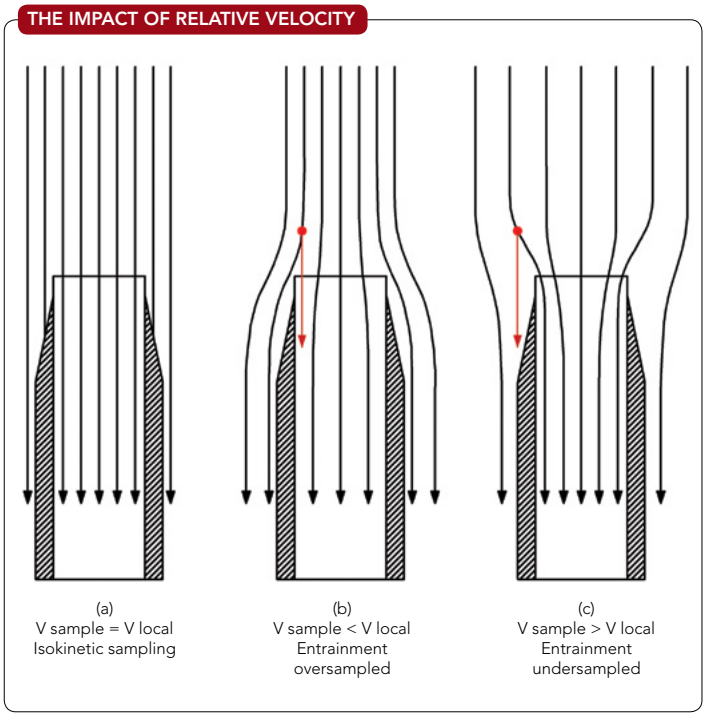

Figure 1. The sample velocity must match the local velocity for accurate results.

Sampling Steam," imposes strict, complex and relatively expensive requirements on sampling. A simpler approach can provide valid process samples. However, similar care is necessary.

Getting accurate process samples hinges on two aspects of sampling: the sampling device and its installation.

The device must be an isokinetic sampling port or equivalent engineered equipment. As long as particle size is small, you readily can adapt standard steam sampling equipment for many process streams. The sampling nozzle size must significantly exceed the particle size you expect to collect. This will minimize problems with entry of particles into the nozzle.

The device must be installed along the axis of flow. It also must be at a location where the local velocity is the same as the average pipe velocity. Figure 2 shows the effect of Reynolds number (velocity), sample location and flow pattern development. A sample point 0.2 pipe diameter from the wall is a nearly ideal location. For turbulent flow, this location gives a local velocity that is very close to the average velocity across a wide range of conditions. Longer straight piping that runs both upstream and downstream makes sampling more reliable — whenever possible, provide straight pipe for 15 diameters upstream and 10 diameters downstream.

Installation tends to be simpler for sampling entrained solids. Solid particles will contact the pipe and sampling equipment walls but, as long as they're not sticky, they bounce back into the gas stream.

Liquids present more problems. Pipe bends create areas where inertial forces tend to concentrate liquid toward the outside of the bends. The best solution for sampling liquid droplets is to have as few changes of flow direction as possible between the upstream separator (usually a drum) and the sampling point.

Sampling methodology is another key factor. With intermittent sampling, the velocity in the sample port goes from zero to the same velocity as the line and then back to zero. So, it's important to only collect a sample when the sample port velocity is the same as the line velocity. If this can't be done, take different-duration samples to allow for corrections to account for the changing velocity profile during the sampling.

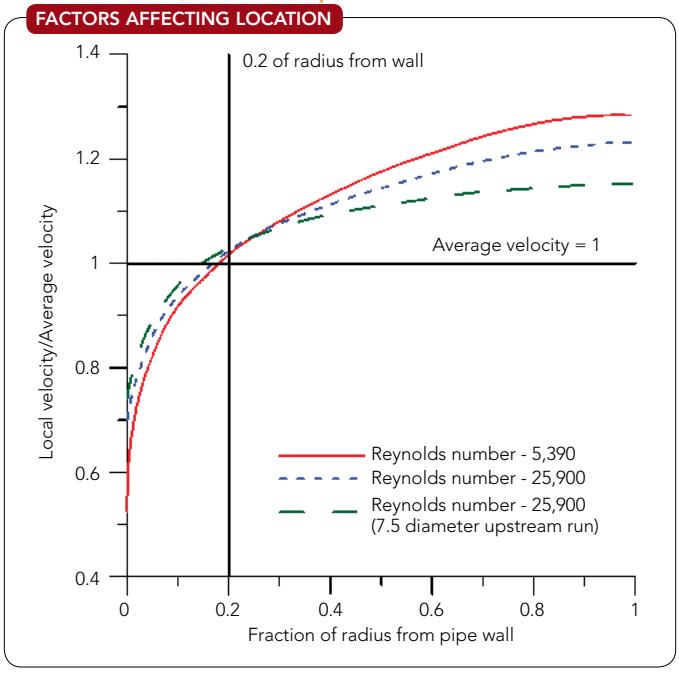

Figure 2. A sample location 0.2 pipe diameter from wall generally is ideal.

ANDREW SLOLEY, Contributing Editor
ASloley@putman.net

Conquer Pilot Plant Challenges

Use design of experiments to minimize the number of runs and ensure meaningful data

By Ron Stites, Stites & Associates, LLC

A PILOT plant usually plays a key role in process development by providing essential data related to operation, safety, scale-up and other issues. Of course, the value of the pilot plant depends upon the validity of the data captured. Planned experiments are crucial to gathering meaningful data. Identifying variables in advance and controlling them during runs must underpin efforts.

Changing one factor at a time rarely is effective because it requires a large number of runs and fails to reveal factor interactions. On the other hand, managing multiple factors demands discipline. Thinking about interactions between factors encourages thorough upfront research and consideration of actual causality, which frequently is 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 to determine whether or not the results are statistically significant.

Whenever possible, randomize the manipulation of factors. The example shown in Figure 1 demonstrates why this is important. Let's suppose we're testing a catalyst for making product at

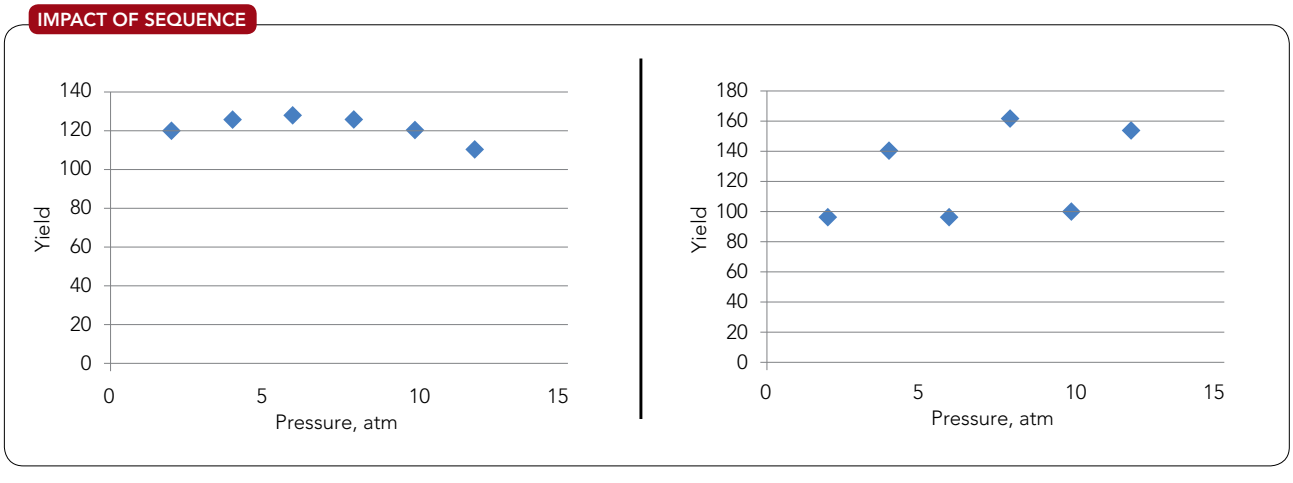

Figure 1. Runs covered in the left graph used progressively increasing pressure while those in the right graph randomized the order of the pressure.

various pressures. In this case, it's a lot easier when running experiments to start at low pressure and then progressively raise the pressure. Ordering the experiments this way provides the smooth curve shown in the left graph in Figure 1 and leads us to conclude that the optimum yield occurs at around 6 atm. What we didn't realize was that catalyst activity was decreasing with time. Running the test in random order of pressure gives the graph shown on the right of Figure 1. It clearly indicates that one or more factors besides pressure are playing an important role.

REAL-WORLD EXAMPLES

Let's now look at several actual applications of DOE in pilot plants to quantify the effects of factors, singly and in combination, while ensuring the statistical validity of the results. All these examples used Design-Expert software from Stat-Ease, Inc.

Our first case involves a pilot plant designed to study carbon sequestration using an organic compound to absorb CO_2 from flue gas. During early runs, a white precipitate formed under certain conditions. Engineers used DOE to manage a series of tests aimed at revealing under what conditions the precipitate would form. The runs showed that controlling the amount of water used in the reaction could eliminate the precipitate. Furthermore, optimizing the water content also boosted the capability of the process to capture CO_2. The result was a significant increase in the performance/cost ratio of the new process.

The second example relates to a pilot plant to optimize an oil-mist recovery system for improving the efficiency of a large power plant. A literature search revealed that product collection most likely depended upon velocities and pressure drops and was nonlinear in its response. However, raising the pressure increases the amount of energy required to operate the recovery system, which, in turn, boosts operating costs. DOE was used to

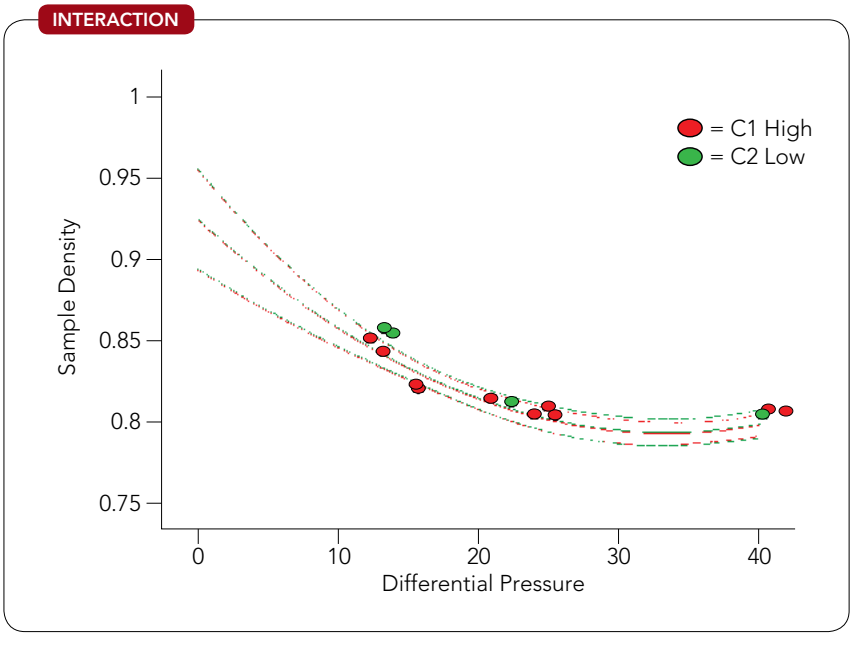

Figure 2. Design of experiments shows a nonlinear relationship between differential pressure and sample density in oil-mist recovery system.

model the sample density (the inverse of product recovery) as a function of the differential pressure and the spray volume. As Figure 2 shows, the results identified interactions between the factors that would have made optimizing their values impossible by testing each variable in isolation. With DOE, engineers found the lowest possible pressure drop that provided a suitably high yield while running the minimum number of tests.

Our third case focuses on evaluating a new catalyst for a process to convert syngas to alcohol that could be used as fuel. The pilot plant had to assess many factors — e.g., the H_2/CO ratio, temperature, gas hourly space velocity (a measure of how fast gas flows over the catalyst) and the proportion of gas recycled. Optimizing alcohol yield was the key goal but other issues included the proportion of methanol to ethanol, tail-gas composition and the rate at which the catalyst degrades. DOE optimized the performance of the catalyst and led to 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.

Our last example illustrates the downside of ignoring DOE. A company developing a device to improve ethanol yield performed trials without using DOE at several operating plants. Because of the promising results achieved, the firm brought the device to market and sold several large systems.

Unfortunately, their performance didn't measure up to the trials. So, at this point, the company re-ran the tests using DOE and discovered the original positive results couldn't be reproduced and most likely were just statistical noise. The designed experiment showed the new device actually had a negative impact on the process. The vendor abandoned the market at considerable expense but DOE saved the firm from wasting additional money trying to reproduce the original positive results. As this application demonstrates, poorly designed and executed trials can have very negative consequences.

PROFIT FROM DOE

Pilot plants are expensive to operate, so it's important to gain understanding from them in the minimum amount of time. In addition, ensuring the insights gleaned 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 determine the relationship between the factors and the responses. At the same time, DOE provides the rigor that establishes benefit/risk relationships with statistical validity, giving the precision and accuracy needed for making decisions about commercial plant investments.

RON STITES is managing member of Stites & Associates, LLC, Brighton, Colo. E-mail him at ron@tek-dev.net.

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Case Study: Sampler Helps Control Process Safely

Automatic samplers can be key to ensuring process samples are repeatable and reliable.

By Sentry Equipment Corp

RUNNING AN efficient operation requires analysis of controlled, real-time data that can be obtained only through reliable and repeatable representative sampling within the production environment. Only with accurate data monitoring and measuring can operations managers obtain the critical insights needed to control and optimize a plant's processes.

Manufacturing and quality control engineers in the chemical industry are faced with the daily challenge of maintaining regulatory compliance using questionable sample analysis results. This is especially true for production environments where industrial waste volumes must be accurately monitored and characterized. Standard sampling practices are not always enough for complete confidence in the results.

CUSTOMER CHALLENGE

To meet stringent regulatory requirements using validated methods for industrial waste effluent, extra effort must be made to ensure the composite sample is reflective of the actual flow of the sample stream. When a composite sample is used for a flowing stream, particularly a toxic waste stream, volumetric accuracy and repeatability is critical, so measures must be taken to assure the sample amount and volume are accurate and repeatable.

This was the challenge for a pharmaceutical company working with a sample stream containing both liquids and solids, otherwise known as slurry. This is a classic application for the Sentry Isolok SAA automatic fixed volume sampler (Figure 1). However, this customer's particular application required:

- Double validation for each sample. The sampler controller actuates a solenoid to cycle the sampler, which, in turn, extracts the sample from the process stream. Typically, composite sample calculations and control are made by simply multiplying the solenoid actuations by the volume of the sampler annulus. In this particular case, this calculation method was insufficient for the customer application.
- Direct indication of each sample completion to fully validate the composite sample volume related to effluent flow, and the customer required positive validation that the sampler fully actuated and extended into and retracted from the process stream.
- An ATEX Zone 2 design (similar to NEMA Class I, Division 2) for a hazardous sample environment.
- The ability to handle a highly corrosive slurry sample, which would necessitate special alloy construction.

AUTOMATIC SAMPLER

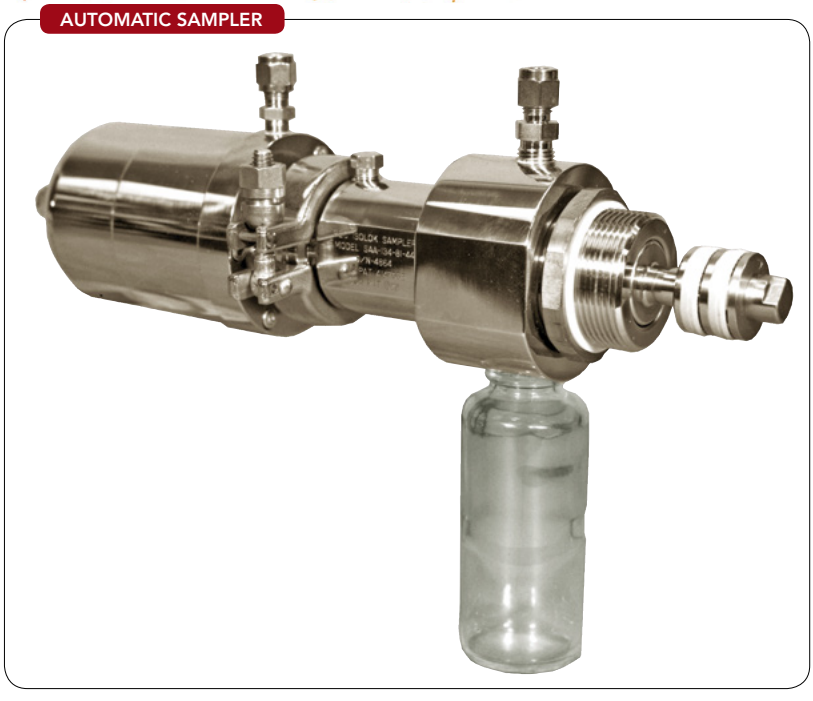

Figure 1. This automatic automatic fixed volume sampler can handle a sample stream containing both liquids and solids.

APPLICATION SOLUTION

To meet the specific application for this pharmaceutical chemical customer, Sentry developed a unique design of its Isolok SAA automatic fixed volume sampler.

The double validation of the sample stroke called for verification of both the extend and retract functions of the sampling process to have a direct indication of each sampler stroke. Obtaining a direct indication of both the extend and retract positions of the sampler proved to be a bit more complicated than originally thought.

While the standard 316 stainless steel actuation cylinder has the ability to validate the retract position to assure the sampler probe is out of the line, it does not have the same ability for the extend function.

A standard aluminum pneumatic cylinder could typically handle this; however, aluminum reacts with caustic and corrosive chemicals, and is not allowed in this type of ATEX hazardous environment.

To meet requirements, the Isolok SAA sampler was custom designed with a 316 stainless steel actuating cylinder with embedded magnetics, along with external ATEX-approved proximity switches mounted on the cylinder. As straightforward as this would appear, this design had to comply and be built to stay within compact cylinder space tolerances. All wetted parts of the sampler used high nickel alloy C-276, a normal variation for the SAA sampler, while all other metallic components were manufactured using 316 stainless steel for corrosion resistance.

The design of this automatic fixed volume sampler for slurry allows for direct indication and double validation of sample volume. The sampler's compact stainless steel actuating cylinder is suitable for sampling any chemical application.

In addition, because it provides a validated number of sampler strokes and the known sampler annulus volume, the sample data it provides allows for a statistical analysis of any degradation of the sampling process from issues such as seal failure, line blockage or erosion of the sampler annulus.

POINT SAMPLER

Figure 2. This point sampler can take a representative sample from hoppers or drop chutes, pressure or vacuum conveyor lines, air slides and moving conveyor belts.

Operations managers and engineers seeking confidence in their sample analysis of hazardous materials for regulatory compliance know it all starts with a reliable, representative sample. This case study highlights how the Isolok SAA automatic fixed volume sampler provides just that, repeatedly.

CHEMICAL SAMPLING SOLUTIONS

You can't control your process if you don't know what you don't know. Automatic samplers can be key to ensuring process samples are repeatable and reliable, every time.

In addition, to delivering true representative sampling and analysis techniques, automatic samplers enable chemical manufacturers to accurately monitor and measure processes for improved production efficiency, output and safety.

Automatic samplers such as those from Sentry can handle chemical sampling solutions for:

Liquids and slurry: Samplers built for pipes, tanks, mixers and reactors can sample any liquids or slurry, whether toxic, corrosive, high temperature or high pressure applications.

High viscosity liquids and gels: Equipment can sample liquids and gels up to 150,000 cP.

Powders, flakes and granules: Point, strip or crosscut samplers (Figure 2) take a representative sample from hoppers or drop chutes, pressure or vacuum conveyor lines, air slides and moving conveyor belts.

Sanitary materials: Several sampling solutions are suitable for any sanitary applications.

With proven sampling expertise since 1924, **SENTRY** products and services provide business operations the critical insights to optimize process control and product quality – delivering true representative sampling and analysis techniques to chemical customers around the globe. For more information, contact sales@sentry-equip.com.

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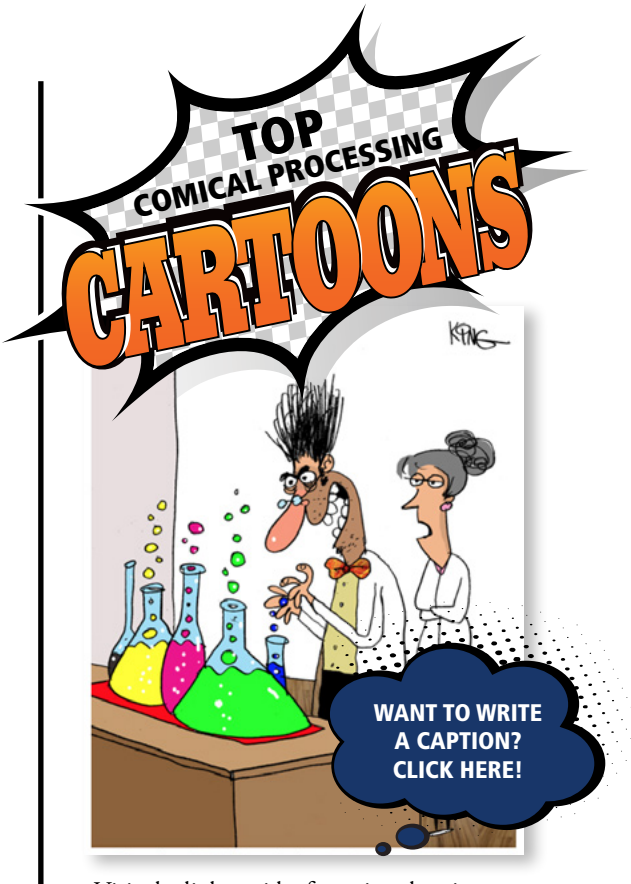

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