Sampling and online analysis tools

Quality control and quality assurance are important systems in delivering the desired product to the customer

by Mark Fowler

Quality control and quality assurance are two important yet distinctly different objectives. As defined by Dictionary.com, quality control is a system for verifying and maintaining a desired level of quality in an existing product or service by careful planning, use of proper equipment, continued inspection and corrective action as required. The definition for quality assurance is a system for ensuring a desired level of quality in the development, production or delivery of products. The former involves verifying the quality of the products produced while the latter is ensuring the process to deliver a quality product.

The difference is reactively testing the product versus proactively managing quality production. Both quality control and quality assurance are important systems in delivering the desired product to the customer. However, that is not the purpose of this article. A common denominator in either system is product sampling.

Laboratories spend countless hours and significant expense to calibrate analytical equipment, train laboratory personnel and assure the accuracy of the results from the testing performed in their lab to measure quality characteristics. The sometimes overlooked aspect of the quality assurance or quality control analysis is the quality of sampling. While the accuracy of laboratory analysis can be well documented, the quality of the test result is only as good as the quality of the sample tested. Proper sampling protocols must be defined and implemented to ensure a representative sample is collected which ensures the validity of the tests.

REPRESENTATIVE SAMPLE

How is a representative sample defined? A representative sample has quality characteristics equal to that of the entire lot or batch tested. Obtaining a representative sample from a product process as large as a flour mill requires consistent, accurate and repeatable methodology. Defining and implementing a standard protocol for sampling ensures consistency. Samples that are not collected in the same way will not represent the process sampled.

Use sampling techniques that have as little sample bias as possible. A simple definition of sample bias is the exclusion of a portion of the product. One example is taking a sample for the testing of break release. The sampling technique must ensure the entire distribution of product ground on a particular roll is collected. If the sample collection technique is flawed, the sample may be biased, albeit unintentionally.

When determining an effective sampling protocol, first profile the process. What is the process variability that is being tested? Do you need a continuous sampling system or is a random grab sample sufficient? Whatever the decision, the sampling system or protocol implemented must reduce sample to sample variation and sample methods and results must be repeatable. You must be assured variability in test results is not the result of sampling variability.

OBJECTIVES

There are several objectives to testing. As previously stated, the most basic objective is to collect a representative sample of the product to be tested to ensure the validity of the testing. Another objective is to collect information. When applied to a quality assurance process, sampling is done to collect internal information on product mix characteristics, such as moisture, ash and protein content of flour in process, or moisture and protein of incoming wheat. The objectives are to manage process variations and maintain process control. Ultimately, consistency is the quality characteristic that customers most value. It will never be listed or defined in a contract, but if the contract asks for minimum 11% protein content, be assured, the customer will be happiest with a consistent supply of 11% protein flour as opposed to deliveries that vary even on the high side.
When applied to a quality control process, sampling is done for the customer. In most cases, the customer receives a Certificate of Analysis (COA) with each shipment of flour. We need to have some assurance in the process in which the sample taken and tested, to ensure the sample represents the load of flour delivered to the customer. In this case, the objective of sampling is product identification. Was the correct flour loaded and shipped to the customer? The basic objective is still to collect information.

TECHNIQUES

How do you collect a representative sample? There are several techniques applied to sampling. To apply the correct technique a good understanding of the material properties of the product being sampled is vital. In our process, we are primarily working with granular materials or powders. Separation occurs in samples. Mixing the sample prior to testing is recommended. A “grab” sample or random sample is often collected in the mill once every couple of hours. This is only a spot check of a very small, statistically insignificant portion of the process, but if the process has been operating within the expected parameters or “in control,” the sample can represent the flour produced over a period of time.

In some cases, a continuous sampling procedure and analysis procedure is preferred. The most common example is automated grain conditioning systems. The flow rate and moisture content of the clean wheat delivered mixer is continuously sampled and tested. The feedback or result is sent immediately to a controller to adjust operating parameters, in this example the amount of water added, to ensure the target moisture content of wheat to the milling system is achieved.

Batch sampling is the most common technique for the quality control protocols to issue a COA to the customer. To fulfill your requirements, you need that final sample collected while loading a bulk vessel or packing flour into bags to analyze the finished product and communicate to the customer that the product meets its desired specifications. In our milling operations, there are several options for taking representative samples. The most common sample location in the mill, especially for grab samples, would be your many gravity spouts.

For incoming wheat or outgoing finished product, a grab sample can be taken from the top of a bin, rail car or truck using scoop or probe. A more representative sample could be obtained using a continuous sampler that takes a series
of small samples to create a composite sample representing the flour loaded or wheat unloaded.

CONTINUOUS SAMPLING PRACTICES

If it is determined that continuous sampling is preferred to obtain a composite sample, the next step is determining the proper sampling device. The proper sampler is determined, in part, by the sampler location. To determine the best sampler location, accessibility is only one concern. The preferred location for the sampler is in a location where the product flow allows for a representative sample to be taken. Sample frequently needs to be often enough to identify process cycles, but the sample size must be manageable. The sampling tool is critical for providing a representative sample. The sampler must assure the sample integrity is preserved. A continuous sampler can also be used to keep the operator safe from any process hazards that may be present.

There are three basic classes of samplers for bulk solids such as grain and flour: point style samplers, strip style and cross cut samplers. A point sampler takes a sample from a point in the material stream. It is best used when material is homogenous such as flour or grain. It is not ideal for a ground product with variability in density, particle size and composition. A strip sampler collects a sample in pneumatic lines or gravity spouts. Strip sampling is a good option when the material in the line may not be homogenous. Ground stock or stock with a wide particle size range is a good example of stock that would be best sampled using a strip sampler. A cross cut, sometimes referred to as a pelican sampler, takes a cross-section of the entire product stream. Crosscut samplers are designed to be used in gravity chutes, and vertical or angular spouts. This sampler style provides the most representative sample, but requires a lot of headroom for installation. It is the most common sampler used for continuous sampling of grain during loading. It

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**GATHERING INFORMATION**

Sampling is the process of gathering information. The objective is to collect a sample that represents the quality characteristics of the entire quantity being sampled. The proper sampling technique requires a defined protocol to eliminate sample variation. Determine the appropriate size of the sample. Too small a sample may not be representative or large enough to conduct the necessary tests. Too large a sample may result in sample bias due to sub-sampling in the lab before testing.

The purpose of sampling and testing for quality assurance is to help identify variation in process control. The purpose of sampling for quality control is to ensure the correct product is delivered to the customer. The fundamental principle in several continuous improvement process strategies is that you cannot improve what you cannot measure. It is also true to say that you cannot measure what you cannot effectively sample.

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November 2013 / World Grain / www.World-Grain.com