Auditing Your Lab to Insure Sensor Reliability

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ABSTRACT

There is a heavy reliance on well calibrated sensors in today’s Pulp and Paper mill. The sensors are feeding critical information to automated control systems. Most sensors require the establishment of a relationship between the raw signal and the laboratory test. Any laboratory repeatability and accuracy issues are transferred to the sensor. This will degrade the sensor performance and the confidence in its result even if the sensor is mechanically and electrically sound. It is important to measure the variability of both the lab and the sensor before any attempt is made to correlate to the sensor. No sensor can correlate any better than the combined lab and sensor variability. Most manufacturers report the sensor accuracy and repeatability in their product brochures. However, laboratory repeatability will differ from mill to mill and should be quantified before calibration begins. Laboratory test results are made up of the components:

\[
\text{Variance}_{\text{total}} = \text{Variance}_{\text{process}} + \text{Variance}_{\text{measurement}}
\]

Our goal is to estimate the various components before the calibration of a new sensor begins. The mill must agree on what is an acceptable measurement error beforehand. Large measurement error can be reduced through improvement of the mill’s testing procedure, laboratory equipment, examination of chemicals used in the tests, and adequate training of testers.

This paper will highlight the importance of good sampling procedures and laboratory methods. Examples of laboratory audits will be presented and how specific issues were addressed.

INTRODUCTION:

Mills have increased their dependence on in-line sensors and semi-automatic analyzers to rapidly update the operators and the control algorithms in the DCS. It should be noted that the output of most sensors are a mathematical relationship between a laboratory test and the sensor’s raw signal output. The sampling procedures, laboratory methods and equipment used will become very important to the overall accuracy and precision of the laboratory test. An inaccurate lab test will lead to an inaccurate output of the sensor. The capital investment in new instrument will not yield optimal performance and the projected benefits even if the instrumentation has been installed properly and well-maintained.

No sensor can perform any better than the combined laboratory and sensor variability. Measurements have two very important properties: Precision and Accuracy.

- **Precision** describes the amount of spread or variation between successive measurements on the same item.
- **Accuracy** is a measure of “how close” the measurement is to the actual property we are attempting to quantify.
It is quite difficult to measure accuracy since it is rare that we know the “real” value of the item we are sampling. Instead, we attempt to quantify the Repeatability and Reproducibility of the measurement.

- **Repeatability** is a term used to describe a precision study for an individual operator’s variation when the same sample is tested under the same conditions.
- **Reproducibility** is a component of precision associated with operator-to-operator differences when several operators run the same sample under identical conditions.

Laboratory test results are made up of the components:

\[
\text{Sample Variance} \quad \text{(total)} = \text{Variance} \quad \text{(process)} + \text{Variance} \quad \text{(measurement)}
\]

The goal of proper sensor calibration is to estimate the various components and review this information before the sensor is placed into service. The mill and instrumentation supplier should agree on the acceptable measurement error before any attempt is made to correlate the laboratory to the instrument. Large laboratory measurement error can be reduced through improvement of the mill’s sampling equipment, sampling collection methods, test equipment, testing procedures, examination of chemicals, and training of testers.

Statistical Process Control provides a valuable tool to maintain sensor uptime and consistent results by helping to establish a systematic approach to evaluate sensor and laboratory performance. This paper will discuss the steps to critique laboratory procedures and to quantify the measurement error from tests used to calibrate instruments.

**THE IMPORTANCE OF PROPER SAMPLING**

A good sample valve is critical to obtain a representative sample from the process. Some mills elect to use a ball valve already installed nearby to save on the cost of the sample valve and its installation. There are several problems when using a ball valve for sampling:

1. Sample is taken at pipe wall
2. Dewaters pulp unless fully opened
3. Different opening degrees at different occasions and for different operators
4. Gives a large volume and splashes at high line pressure.
5. Often plugged leading to a dangerous condition when the field operator is required to clean out the sample line

Is the cost savings worth the bad result? The valve can be throttled and give a misrepresentation of what is the process pipe, especially when sampling for a consistency test. The cost savings will lead to a non-representative sample that will be processed and used in the calibration of the sensor.

![Figure 1: Operational differences between a ball valve versus a sampling valve](image)
A good sampling valve will ensure that a representative sample will be collected. The characteristics of a good sample valve:

1. Takes the sample well inside the pipe wall – clears the water layer
2. Always fully open
3. Not depending on operator
4. Safe even under high pressure and temperature

It is important to pay attention to the installation requirements of the sampler. Line pressure fluctuations can change the laboratory result as shown below. An engineer was having difficulty calibrating a consistency sensor at the beginning of the bleach plant. The line pressure was changing (not known at the time) and samples had been taken at various line pressures. The difference between the laboratory result and the sensor output changed dramatically as shown in figure 2.

Sample valve manufacturers always include a graph that shows the line pressure needed to accurately measure a certain consistency for hardwood and softwood. Figure 3 is an example from a manufacturer’s product sheet.

Line pressure should be measured before selecting the sample valve to ensure you are purchasing a valve that will perform in the consistency range you need.

Sample handling is equally as important as the selection of the sampling valve. A common practice is to collect the sample in an open bucket or separate open containers and take the sample back to the lab for processing. This is detrimental to obtaining an accurate consistency measurement as the wet weight will be changing due to the evaporation of the sample in the open container. A better method would be to cover the bucket or the individual containers. A chlorine dioxide sample for residual analysis will gas off if the container is uncovered. The sample is not representative of the process conditions and is “bad” before it arrives at the laboratory.
In a North American mill, the consistency sample was collected in an uncovered bucket and walked from the field to the laboratory (1500 feet in distance). Once the tester returned to the laboratory, 3 samples were taken from the bucket and analyzed immediately. The sensor was calibrated in this manner and had been thought to give excellent results with low differences between the sensor and the laboratory. It was suggested that the tester cover the bucket after the sample was collected. The difference between the sensor and laboratory result increased greatly as shown in figure 4. It appeared that the original method gave consistency results higher due to the change in the wet weight of the sample (water was driven off in the open bucket). The result was an over estimation of the consistency. The tester elected to recalibrate the consistency sensor using the covered bucket method in order to obtain more realistic results.

![Range Chart for Lab versus Consistency Transmitter](image_url)

**Figure 4: Impact of changing sample collection procedures**

**VARIABILITY MAKERS IN THE LABORATORY**

A good sample valve and proper method to get the sample to the laboratory will reduce the overall variability in the test result. However there are many other sources of variation encountered when running the pulp sample for the standard pulp tests such as kappa, brightness, residual, pH and consistency. Some common problems encountered are:

1. Sub-sampling from the bucket. Did we adequately stir the pulp between sample collections?
2. Container was not properly weighed. Was the container dry before we weighed it empty and added our pulp?
3. Sample is not well-washed.
4. Variable pad drying time. Is the pad truly dry?
5. Was the dried pad weighed hot or cooled in a desiccator before weighing?
6. Are we using filter paper or is the pad made directly on the sheet former.
7. Typing of drying (speed dryer versus air drying)
8. Test equipment not calibrated properly (scales, titrators),
9. Is the testing equipment clean and free of contaminants (previous test pulp not completely removed).
10. Testing chemicals (purchased strength needed for the test or made in the mill’s laboratory).
11. Is temperature compensation used when necessary?
12. Improper measuring of test chemicals.
13. More than one tester involved in the calibration. Does everyone do the test the same way? Has there been proper training in the test method?

Each item mentioned will add error to the measurement under evaluation. Mills have an expectation that the difference between the laboratory result and the calibrated analyzer result will be very small. In this example, the mill’s expectation was to see no more than a 15 point difference between a lab check and the on-line freeness analyzer. The analyzer was published to have a repeatability of $\sigma = 5$ ml CSF (at 400 ml CSF). The initial results showed much larger deviations. The mill and the vendor worked together to find the cause of the larger deviation. The testing procedures were reviewed and the average difference between repeated freeness samples was 25. The tests were used to construct a 3 sigma limit Repeatability Range Chart. The Range is defined as the difference between the maximum and minimum of a set of numbers. The Range was used to calculate an Upper Control Limit showing that under normal testing conditions, the lab tests could differ as much as 65 points!

![Lab Freeness Repeatability Chart](image.png)

Figure 5: Improving Lab Repeatability
Issues were found with the sampling collection method and the scale used in the testing. This affected the consistency used in the freeness calculations. Figure 5 shows the starting differences between repeated lab tests and the later improvement in June by pinpointing and resolving issues with the freeness testing method.

CONDUCTING A LABORATORY AUDIT

What is the best way to tackle variable makers and reduce their impact on the performance of in-line sensors and analyzers? What is considered “small” difference between the laboratory and the sensor? This difference is dependent the laboratory factors previously mentioned and the training of the personnel. As in the previous example, the mill wanted a small difference between the laboratory freeness and the analyzer freeness. This was not possible under the current laboratory conditions. The first step is to perform a laboratory audit that will identify issues that are introducing error into our lab result. This begins by involving all the testers responsible for testing and calibration.

The best situation for calibration is to have only one tester run all the samples to be used in the model and estimate the tester’s repeatability. This is not always possible so it becomes important to estimate the repeatability of the all testers who may run a test during the calibration and validation process. We begin by taking 30 sets of samples and analyze them according to the current procedure. Each tester would analyze 4 to 5 sets of samples. This information will allow us to calculate the percent measurement error of the sample. This measurement error is transferred to the sensor calibration, so it is critical to quantify this as it will affect the sensor performance.

In general the procedure is to calculate the Percent Total Variation due to the measurement system by the formula:

\[
\text{% Measurement Variation} = 100 \times \left( \frac{\text{Standard Deviation}^2_{\text{Measurement}}}{\text{Standard Deviation}^2_{\text{Total}}} \right)
\]

\[
\text{Standard Deviation}^2_{\text{Total}} = \text{Process Variance} + \text{Measurement Variance}
\]

Values over 20% indicate that the laboratory methods need improvement. It is not uncommon to see values over 30% in a mill with poor equipment, test methods and training.

Let’s consider an example where the mill was calibrating a kappa analyzer for kappa range 50 to 60. The laboratory had 3 auto titrators that would dispense the various chemical used in the kappa test at the appropriate time. Two titrators were routinely used and the third was kept as a backup in case of failure. The mill was duplicating the sample and running each sample on a different titrator. The sample was obtained through the kappa analyzer’s collection sample. Two separate pads were made and the correct amount of dried pulp was measured from each pad. There was generally a 1 to 3 kappa point difference between pads. This was due to errors in measuring the dried pulp, the dryness of the pulp pad, and condition of each titrator.

A 3-sigma range chart was constructed to show what the normal variability should be between the tests. The Range chart showed that it was possible to get as much as a 4 point difference between the repeated samples and the percent measurement error was normally around 22%. The calculations are shown in the table below:
There was a time period where the difference would be as much as 7, 8 or 9 points which was not normal for the laboratory. Several issues were discovered:

1. Chemical lines from the bulk chemical storage to the titrators that dispense the chemical were partially plugged. The correct amount of acid, potassium permanganate, and sodium thiosulfate was not added during the test cycle.
2. Drying time for each pad was not the same, so the weight of the sample used was not correct
3. The blank was not being run on each titrator per the mill laboratory procedures.

<table>
<thead>
<tr>
<th>Source</th>
<th>Standard Deviation</th>
<th>Variance</th>
<th>Percent of total</th>
</tr>
</thead>
<tbody>
<tr>
<td>Measurement</td>
<td>1.26</td>
<td>1.60</td>
<td>22.25</td>
</tr>
<tr>
<td>Total</td>
<td>2.68</td>
<td>7.18</td>
<td></td>
</tr>
</tbody>
</table>

Differences between the repeated samples improved after the titrators were serviced and the tubing dispensing chemical were replaced. Testers were retrained in the kappa test procedures. Proper procedures were used to run daily blanks through the auto-titrator.

The average difference between the repeated samples was now 0.90 and the percent measurement error was reduced to a little over 11%.
The construction of the lab repeatability chart was an important tool in helping us determine that something was wrong in our testing procedure. Understanding what normal variation is in our testing process keeps us from adding “bad” tests to the sensor calibration and comparing them to the sensor output. In practice the large difference samples would have been averaged and compared to the analyzer result. The analyzer was reading was reading 55.49, the lab tests were 61.6 52.5 for an average of 57.05.

Which test is closest to the “true” kappa? We don’t know. The best action to follow is to quantify the laboratory error before calibrating your analyzer or sensor. A sample exhibiting a large difference between repeated lab samples will be automatically excluded from the calibration. A large difference is one that is above the Upper Control Limit. Another sample should be taken after it is determined what went wrong with the previous test.

CONCLUSIONS:

Today’s manufacturing environment relies heavily on in-line sensors and analyzers. Poor sampling and laboratory procedures will influence the output of the sensor or analyzer. No sensor should be calibrated until the laboratory measurement error is quantified and reduced to its lowest possible amount. The measurement error will be part of the sensor’s overall error. Good sampling collection methods and sample valves are a critical part of the laboratory process. Laboratory equipment and procedures should be reviewed before embarking on a major calibration effort. All testers should be trained to follow the same procedure in order to minimize variability and ensure a solid calibration for optimum sensor performance.

REFERENCES: