Abstract

A distilled treatment of both the beet and cane sugar production processes are presented in a clear and concise format. The discussion includes details on monitoring and control parameters along with installation guidelines and sensor cleaning suggestions. pH sensor maintenance and troubleshooting are covered as well.

Brief History of Sugar Production

Two major sources for the production of sugar for consumption are sugar cane and sugar beets, with sugar cane being both the older and more prolific of the two.

The sweetening properties of sugar cane were known as early as the fourth century B.C. However, refining did not begin until over 1,200 years later in Spain and southern France during the eighth century. In 1493, Columbus brought sugar cane to the West Indies and so began the growth of cane plantations and refineries in the Americas.

Beet sugar, on the other hand, is a relative newcomer in the sugar production industry. The world’s first beet sugar pilot plant began operating in 1802 in what is now Poland. When Europe was cut off from its West Indies sugar supply during the Napoleonic wars, additional research into beet sugar production was stimulated. In 1811, Napoleon established six sugar beet schools, funded one hundred students and built 10 new factories for beet sugar production. During the mid to late eighteen hundreds, beets were the major source for refined sugar worldwide. By the middle of the twentieth century, cane sugar rose to the top and has remained there through the present. Even so, sugar beets still constitute a significant source of raw sugar production world wide. Sugar is grown and produced principally in:

<table>
<thead>
<tr>
<th>Beet Sugar</th>
<th>Cane Sugar</th>
</tr>
</thead>
<tbody>
<tr>
<td>USSR</td>
<td>Cuba</td>
</tr>
<tr>
<td>United States</td>
<td>Brazil</td>
</tr>
<tr>
<td>France</td>
<td>Australia</td>
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<tr>
<td>Germany</td>
<td>India</td>
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<td>Poland</td>
<td>United States</td>
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<td>Italy</td>
<td>Mexico</td>
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<tr>
<td>United Kingdom</td>
<td>China</td>
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<td>—</td>
<td>South Africa</td>
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<td>—</td>
<td>Philippines</td>
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<tr>
<td>—</td>
<td>Argentina</td>
</tr>
</tbody>
</table>
Modern production of sugar from both cane and beets was made possible by the invention of three basic machines: the vacuum boiling pan in 1813, the centrifuge in 1837, and the multi effect evaporator in 1843. pH control is critical throughout these processes, from initial extraction through crystallization.

Introduction

Sugar production is a high-temperature, caustic process requiring tight pH control in almost every stage. Because the process is so physically demanding, pH measurements have typically been made in cooled sample lines (introducing significant lag time) or via grab samples (infinite lag time). Attempting to control a nonlinear variable such as pH in a continuous, dynamic process using the methods described above can easily cause loss of process control. Prior to improvements in both pH glass and reference technology, online measurements were virtually impossible, pH excursions occurred on a regular basis, and shutdowns were not uncommon.

Ideally, pH measurement and control should occur at process temperatures and pressures. Dead time must be minimized. These conditions can be met by placing the pH sensor directly into the main process line. With advances in pH sensor technology, it is now possible to make online, real-time, low-maintenance, cost-effective pH measurements in the sugar mill.

This paper addresses these issues by providing a distilled treatment of the actual sugar production process and the measurement and control of pH. It also discusses pH sensor technology, installation and maintenance guidelines, and includes suggestions on calibration, measurement validation and troubleshooting.

Beet Sugar Production

Beet sugar production begins with the extraction process; continues through purification, filtration and evaporation; and concludes with the crystallization process (Fig. 1).

Extraction

At the beginning of the process, previously washed beets are fed into a hopper which transfers them onto the rotating beet slicer. Cossettes (sliced beet pieces) make their way to the diffuser via conveyer belts. Diffusion is strictly a thermo-mechanical process where the cossettes are washed with warm water and lightly squeezed to release raw juice. The diffuser may be either a diffusion tower or a continuous beet diffuser. Cossettes enter the diffuser from below and are carried upward where they are released as wet pulp to the pulp presses. Entering at the top of the diffuser and flowing counter-current to the cossettes is both fresh hot water and recycled press water. This mixture exits at the bottom of the apparatus as a sucrose-rich diffusion juice, usually referred to as raw juice. This extraction is carried out at 71 to 77 degrees centigrade. Formalin may be added to the recycled press water. The addition of formalin in conjunction with high temperature inhibits bacterial growth. The pH at this point should be approximately 5.5 to 5.6 and is usually controlled by sulfur dioxide addition (Fig. 2).

First Carbonation

In the purification and filtration portion of the process (Fig. 3) raw juice from the extraction stage is mixed with milk of lime, Ca(OH)$_2$, to coagulate the impurities which insures
both good settling properties and filterability. The mixture is then heated to 76 to 82 degrees centigrade and carbonated via CO₂ gas injection in the precarbonation tank. The pH is then adjusted between 8.8 and 9.0 with calcium hydroxide, Ca(OH)₂. The juice continues on to another lime addition tank. All carbonation is performed by introducing carbon dioxide gas into the bottom of each tank. Carbonated juice then overflows at the top of the tank. The juice goes from the precarbonation/liming loop on to the first carbonation loop. Juice exiting first carbonation should be in the neighborhood of 11.2 pH. The temperature is typically 88 degrees centigrade. Carbon dioxide saturation at the tank exit indicates the end point of the reaction.

Second Carbonation

The carbonated juice and calcium carbonate precipitate are sent to the thickener or mud-settling tank where the two juices (clarified and muddy) are separated. The muddy juice is filtered and this filtrate and the clarified juice are blended and filtered through ceramic filters. This filtrate is heated to 97 to 99 degrees centigrade, then fed into the second carbonation tank. Once the second carbonation is completed (pH = 8.7 to 8.8, temp. = 98 degrees centigrade), the juice goes to a collection tank where it is again filtered. Finally, the juice is injected with sulfur dioxide (sulfitation), a bleaching agent that lowers the pH to around 8.0, and is then sent to the evaporators.

Figure 1. Beet Sugar Process Overview
Evaporation

The pH (8.0 to 9.0) is checked at the inlet to the evaporator section where the juice will be concentrated down to a thick juice which is then mixed, in the crystallization portion of the process, with remelted liquor from second sugar and filtered to become the standard liquor. At this point, near the end of the process, we have the makings of two intermediate products and the final product, white sugar.

Crystallization

To make the white sugar product (Fig. 4) standard liquor is continuously fed into the vacuum pans, which have been previously seeded to promote crystal growth. Here, pH is controlled at 8 to 9 pH with sodium hydroxide, NaOH. The temperature at this point has dropped to the 70 degrees centigrade range. Sugar crystals are grown in a mixture of standard liquor and first molasses. This massecuite (standard liquor, first molasses and suspended sugar crystals) is centrifically separated into raw sugar and molasses. The molasses is then reboiled to obtain the second massecuite and again separated into the second raw sugar and second molasses. This process is repeated a third time to obtain the third sugar and third molasses. Exhausted (third) molasses is removed from the process via the exhausted molasses tank.
**Figure 3.** Beet Sugar Purification and Filtration

**Figure 4.** Beet Sugar Crystallization
Typically, the sucrose content of exhausted molasses is around 50 percent. In older facilities, the Steffen process is employed for sucrose recovery. The exhausted molasses is diluted, and finely powdered quicklime, CaO, is added to create a saccharate of lime precipitate which is in turn used instead of or in addition to milk of lime in the beet juice liming process. The quality of this mixture is monitored via pH, usually between 8 and 9. The main drawback to using the Steffen process is that both sucrose ($\text{C}_{12}\text{H}_{22}\text{O}_{11}$, sugar) and raffinose ($\text{C}_{18}\text{H}_{32}\text{O}_{16}$, semi-sweet sugar) are precipitated which decreases the quality of the standard liquor and the final product as well - white sugar.

In newer mills, ion-exchange is used instead of the Steeffan process. The thin juice (pre-evaporators) is decolorized and de-ashed in ion-exchange columns to improve the sugar quality and reduce the volume of exhausted molasses generated at the end of the process.

White sugar, the final product, is dried and sent to packaging plants for distribution. This final product from beet sugar processing does not require further processing or refining. Table 1 outlines the various measurement locations as well as identifying the process and normal installation configurations.

<table>
<thead>
<tr>
<th>Description</th>
<th>Sensor Location</th>
<th>pH Range</th>
<th>Temp (°C)</th>
<th>Reagent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diffusion H$_2$O</td>
<td>In-line</td>
<td>5.6</td>
<td>72</td>
<td>SO$_2$</td>
</tr>
<tr>
<td>Pre carb</td>
<td>2&quot; recirc or hot tap</td>
<td>8.8 - 9.0</td>
<td>80</td>
<td>CO$_2$[Ca(OH)$_2$]</td>
</tr>
<tr>
<td>First carb</td>
<td>2&quot; recirc or hot tap</td>
<td>11.2</td>
<td>88</td>
<td>CO$_2$[Ca(OH)$_2$]</td>
</tr>
<tr>
<td>Second carb</td>
<td>2&quot; recirc or hot tap</td>
<td>8.7 - 8.8</td>
<td>98</td>
<td>CO$_2$</td>
</tr>
<tr>
<td>Std liquor</td>
<td>2&quot; recirc or hot tap</td>
<td>8.0 - 9.0</td>
<td>70</td>
<td>NaOH</td>
</tr>
<tr>
<td>Molasses</td>
<td>2&quot; recirc or hot tap</td>
<td>8.0 - 9.0</td>
<td>70</td>
<td>None</td>
</tr>
<tr>
<td>Sulfitation</td>
<td>2&quot; recirc or hot tap</td>
<td>8.0</td>
<td>70</td>
<td>SO$_2$/NaOH</td>
</tr>
</tbody>
</table>

### Cane Sugar Production

Cane sugar production can be broken down into several distinct operations. The first operation is the processing of cane into raw sugar. The second operation is refining. The refinery is sometimes just another section of the cane mill (similar to a pulp and paper mill), but is often a separate entity. This second operation is where the white refined sugar that we purchase in the grocery store is produced. The third operation associated with cane production is the plantation white sugar process. Plantation white sugar is processed at a single site for direct domestic consumption. Figure 5 shows a general overview of a generic cane processing plant.

### Raw Cane Sugar Manufacturing

After being weighed, the cane is transported to cane feeder tables (Fig. 6), where it is then pushed into cane cutters. Cane knives slice the stalks into chips and compact them onto the bed of the cane carrier for delivery into the crushers. Approximately 60 percent of the cane juice is liberated from the cane (maceration) by the crushers (giant pressurized rollers). The crusher juice is then sent on to a clarifier. In some mills, a shredder is used between the cutting and the roller sections. Hammer-mill type shredders reduce the cane down to its fibrous structure to further facilitate the extraction process.

Crusher bagasse (pressed cane) may be dried and sent to a recovery boiler or in more modern facilities, it is first put through a continuous diffuser, similar to the beet sugar
diffuser, to further extract cane juice. This extraction, called lixiviation, is accomplished by passing the bagasse counter current through a combination of fresh warm water (pH = 5.5 to 7.5, T = 75 degrees centigrade) and clarified press water (commonly referred to as lixiviation water). The diffusion juice resulting from the percolation of warm water through the bagasse is mixed with crusher juice bound for the clarifiers. This mixture is called mixed juice draft. To inhibit bacterial growth, lixiviation is performed at 77 degrees centigrade.

Before entering the clarifier, the mixed juice draft is heated to 105 degrees centigrade and treated with milk of lime (calcium hydroxide) to increase the pH to around 7.5 to 7.8. The bottoms, settled mud juice, goes to a vacuum filter where phosphoric acid is sometimes added to enhance the clarification process. The solids are removed as waste from the filters and the liquid is returned to the effluent of the clarifier which contains the clarified juice (clear juice).

Finally, the clear juice is sent to a quadruple effect evaporator where it is concentrated down to about 65 percent solids, raw syrup, then sent on to vacuum boiling pans where it is further concentrated and sugar crystals are grown. Sucrose deposition from the raw syrup onto the seed crystals continues until the massecuite, a mixture of syrup and crystals, has reached a volume of about 80 percent solids to water. Being a semi-batch operation, when this volume is achieved, the first massecuite is removed and sent to the
centrifuges for separation. Raw sugar from the top of the centrifuge goes to a storage tank. Exiting the bottom of the centrifuge, first molasses goes to another vacuum boiling pan producing a second masssecuite. This second masssecuite is sent to another centrifuge that produces second raw sugars, which go to the product storage tank, and second molasses. The second molasses is further processed in a vacuum boiling pan to produce third raw sugars which are used as seed crystals in the first vacuum boiling pan. Exhausted molasses, commonly called cane blackstrap, is removed as waste. Our final product from the raw sugar mill, a combination of first and second raw sugars, will eventually be shipped to a refining plant. Typical pH control parameters appear in Table 2.
Raw Cane Sugar Refining

Raw sugar is fed into a continuous mingler which mixes both the raw sugar and warm syrup (affination syrup) supplied from the centrifuge (Fig. 7). The product of this mixing, magma, is fed to the centrifuge which produces the previously mentioned affination syrup (warm syrup) and affined sugar. Dissolution of affined sugar is carried out at 66 degrees centigrade by adding hot sweet water from the filter presses and fresh hot water in the melter. The raw liquor from the melter is fed into a liming tank, treated to a pH of 7.5 to 7.8 and then carbonated with carbon dioxide gas. This carbonated liquor overflows into a second carbonation stage where the reaction is completed. At this point, calcium carbonate has precipitated, carrying down a significant amount of the colored substances present in the liquor. This thin mixture is about 60 percent decolorized.

Further decolorization is accomplished in the bone ash (tribasic calcium phosphate) decolorizer after previous filtration of the thin liquor. In many facilities, the bone ash decolorizer has been replaced with a lower cost process using carbonation and an activated carbon exchange device. Ion-exchange is commonly used in Japan instead of bone ash for the final decolorization step. Once the filtrate has passed through the bone ash decolorization stage it is sent as fine liquor to the boiling house for a six to seven step boiling process. Each step produces sugar and molasses, and the molasses is sent to another boiler to undergo yet another round of concentrating and crystallization until the molasses are completely exhausted. Table 3 shows the process parameters for the refining operations.

Products from the refinery include white refined sugar usually made from first, second and third sugars and brown sugar from fourth and lower stage sugars. These products are dried and then either packaged for general consumption or sold in bulk to the food and beverage manufacturing market.

Plantation White Sugar

In South Asian countries, it is common to produce plantation white sugar for domestic consumption. Plantation white sugar is milled in the same way as raw cane sugar, but instead of sending mixed juice draft to a continuous clarifier, it undergoes either sulfitation, a common practice in sugar beet processing (impregnation of sulfur dioxide) or carbonation. All operations are performed at the same site rather than having separate milling and refining locations.

In the sulfitation process (Fig. 8), the mixed juice draft is heated to 74 degrees centigrade and fed into a tank where both milk of lime and SO₂ gas are being introduced. This sulfur dioxide saturated juice exits the talodura (sulfitation) section at temperatures as high as 105 degrees centigrade. The pH here is around 7.0 to 7.5. The juice is treated with phosphoric acid and then the pH is elevated in a liming tank with milk of lime and pumped, as saturated juice, to the clarifier at 7.5 to 7.8 pH. By the time the saturated juice reaches the clarifiers, it has cooled to around 75 or 80 degrees centigrade. In the evaporators, the clarified juice is concentrated into raw syrup which is in turn processed into first massecuite by injecting it with SO₂ gas. The first massecuite is run through a centrifuge where the white sugar product is produced. Data for the pH applications in the sulfitation process are in Table 4.
Alternately, the carbonation process (Fig. 9), may be used to produce the plantation white sugar by heating the mixed juice draft from the cane mill to 55 degrees centigrade and injecting it with carbon dioxide gas and liquid milk of lime in a carbonation tank. It is

Table 3.  Refined Sugar Process Parameters

<table>
<thead>
<tr>
<th>Description</th>
<th>Sensor Location</th>
<th>pH Range</th>
<th>Temp (°C)</th>
<th>Reagent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hot melter H₂O</td>
<td>In-line</td>
<td>5.6</td>
<td>66</td>
<td>SO₂</td>
</tr>
<tr>
<td>Liming tank</td>
<td>2” recirc or hot tap</td>
<td>7.5 - 7.8</td>
<td>70</td>
<td>Ca(OH)₂</td>
</tr>
<tr>
<td>Carbonation</td>
<td>2” recirc or hot tap</td>
<td>9.0</td>
<td>75</td>
<td>CO₂</td>
</tr>
<tr>
<td>Second carb</td>
<td>2” recirc or hot tap</td>
<td>10.5 - 11.0</td>
<td>80 - 90</td>
<td>CO₂,Ca(OH)₂</td>
</tr>
<tr>
<td>Fine liquor</td>
<td>2” recirc or hot tap</td>
<td>8.0 - 9.0</td>
<td>70</td>
<td>None</td>
</tr>
</tbody>
</table>
desirable to control the pH coming out of the carbonation tank between 10.5 and 11.0 pH. The calcium carbonate precipitates are removed and the juice is further carbonated in the second carbonation stage where it is then filtered and sent to evaporators as clarified juice. It exits the evaporators as raw syrup where it then undergoes a sulfur dioxide saturation and filtration before entering the centrifuge as massecuite where it is finally reduced to white sugar. Table 5 shows the pH applications of interest for carbonation.

Figure 8. Plantation White Sugar-Sulfitation

Table 4. Plantation White Sugar Process Parameters - Sulfitation

<table>
<thead>
<tr>
<th>Description</th>
<th>Sensor Location</th>
<th>pH Range</th>
<th>Temp. (°C)</th>
<th>Reagent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mixed draft</td>
<td>2&quot; recirc or hot tap</td>
<td>7.5 - 7.8</td>
<td>75</td>
<td>Ca(OH)₂</td>
</tr>
<tr>
<td>Sulfitation</td>
<td>2&quot; recirc or hot tap</td>
<td>7.0 - 7.5</td>
<td>105</td>
<td>SO₂, Ca(OH)₂</td>
</tr>
<tr>
<td>Liming tank</td>
<td>2&quot; recirc or hot tap</td>
<td>7.5 - 7.8</td>
<td>85</td>
<td>Ca(OH)₂</td>
</tr>
<tr>
<td>Massecuite</td>
<td>2&quot; recirc or hot tap</td>
<td>7.5</td>
<td>70 - 80</td>
<td>SO₂</td>
</tr>
</tbody>
</table>
Table 5. Plantation White Sugar Process Parameters - Carbonation

<table>
<thead>
<tr>
<th>Description</th>
<th>Sensor Location</th>
<th>pH Range</th>
<th>Temp (°C)</th>
<th>Reagent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mixed draft</td>
<td>2” recirc or hot tap</td>
<td>7.5 - 7.8</td>
<td>55</td>
<td>None</td>
</tr>
<tr>
<td>Carbonation</td>
<td>2” recirc or hot tap</td>
<td>10.5 - 11.0</td>
<td>80</td>
<td>CO₂, Ca(OH)₂</td>
</tr>
<tr>
<td>Second carb</td>
<td>2” recirc or hot tap</td>
<td>11.2</td>
<td>90</td>
<td>CO₂</td>
</tr>
<tr>
<td>Raw syrup sulf</td>
<td>2” recirc or hot tap</td>
<td>8.7 - 8.8</td>
<td>85</td>
<td>SO₂</td>
</tr>
</tbody>
</table>
pH Measurement and Control

Most pH control in a sugar mill is accomplished with a feedback loop sending a milliampere signal to a pump or valve for reagent delivery into the process. It is the usual practice to employ only proportional control rather than the more sophisticated PID control schemes. Proportional control works quite well simply because sugar production is a recipe type operation. The cane or beet feed stock is fairly consistent with only small variations in acidity or alkalinity. In many older or third world mills, pH control is completely manual with operators manually opening and closing valves based on grab-sample pH measurements that may or may not employ electronic pH sensors. Some mills actually measure pH with pH paper! The largest obstacle in controlling pH in the sugar mill is finding equipment that will tolerate the harsh conditions found in this type of environment.

With the exception of the carbonation stages in both the beet and cane processes, most of the applications are at neutral or slightly alkaline pH, usually no higher than 9.0 pH. Only the feed water runs in the acidic range (5.6 pH). In the carbonation process, the pH values may run as high as 11.5 in some mills, with most mills running at 11.0 to 11.2 pH. So, for the most part, the pH range is not troublesome as far as having a wide variation in the control parameter.

Temperatures may vary a great deal throughout the process but are usually fairly stable within a unit operation. Most of the time the temperatures run in excess of 70 degrees centigrade and in some cases as high as 105 degrees centigrade (carbonation effluent). As temperatures rise, finding a pH sensor that can survive without substantial degradation becomes increasingly difficult. This is particularly true of the glass measuring electrode portion of the sensor.

Pressures do not usually play an important factor in the measurement and control of pH in the sugar mill as they are not widely variable nor excessively high.

The largest factor adversely effecting pH accuracy, stability and life span is the density of the liquors and the particulate content. Since pH is measured with a sensor that has a glass measuring electrode and porous liquid junction, coating and breakage are of ultimate concern. Usually the goal of the operators and maintenance technicians is to have a pH sensor which will last an entire campaign and require only minimal service and cleaning.

Advances in pH Sensor Technology

Online pH sensors have come a long way since the early to mid seventies. Improvements in both glass and reference technologies have allowed users to make more accurate, real-time assessments of the process pH while increasing the maintenance intervals required for these devices.

Recent improvements in glass technology have allowed users to measure and control pH in very high temperature environments. Many manufacturers can tolerate temperatures as high as 120 degrees centigrade. TBI-Bailey pH sensors will function at 140 degrees centigrade...the highest temperature rating in the industry. This makes TBI-Bailey sensors an ideal candidate for those high temperature applications in the sugar processing industry such as the sulfitation or carbonation operations where the temperatures are consistently above 100 degrees centigrade.
Because of high temperatures and harsh chemical reagents used in the sugar industry, the pH sensor reference will suffer a great deal. Most pH sensors fail because the reference becomes electrically isolated from the process fluid due to plugging or the reference becomes poisoned by the process fluid. Poisoning occurs because the reference electrode is submerged in KCl electrolyte fluid that is in physical contact with the process fluid via a porous liquid junction (Fig. 10). As process fluid works its way into the reference, the chemical characteristics change, chemical reactions between the process fluid and the electrolyte occur, and the voltage potential of the electrolyte changes. This manifests itself in the form of pH drift. To address this drift problem, TBI-Bailey patented the Solid State Reference (Fig. 11), which prevents poisoning of the KCl electrolyte fill by saturating a solid substrate with the electrolyte. The result is a solid substance that significantly slows down the migration of process fluid into the reference area of the sensor. Plugging, on the other hand, occurs when the process fluid adheres to the surface (or just below the surface) of the liquid junction area. TBI-Bailey pH sensors have a very large, annular liquid junction which presents a much larger surface area to the process. This larger surface area resists plugging more readily than a standard liquid junction. These sensors can last an entire campaign!

![Figure 10. Typical Combination pH Sensor](image)

In 1996, TBI-Bailey introduced its Next Step Solid State Reference. This new technology (patent pending) is a significant improvement over TBI-Bailey’s already superior Solid State Reference. New materials have been introduced into the reference area that not only provide better sealing, but also cushion the measuring electrode so that internal breakage is minimized. When it comes to lifespan and accuracy in harsh environments such as sugar mills, no other pH sensor on the market today can compare to TBI-Bailey.

**Installation Guidelines**

Installation of the pH sensor is critical for two reasons. First, the sensor must be located somewhere where it sees a representative sample of the process fluid without introduction of extra lag time. Ideally, a representative sample is at the operating pressure, temperature and flow rate. Since temperature can effect pH via both Nernstian effects and by actual chemical changes in the process fluid due to temperature changes, it is critical that the pH measurement happen at operating temperatures. It is also critical that the measurement be made after reagent addition and after the resulting reaction has taken place, but not so far down stream that it is measuring yesterday’s news.
Second, the sensor must be located in an area that allows for good flow past the sensor. If the process fluid is flowing by the sensor at 90 degrees and at a decent flow rate (three to five feet per second is ideal), the process fluid will actually act as a cleaner (Fig. 12). The wiping action will slow down the deposition of coating materials on both the measuring electrode and the annular liquid junction area. Less coating means better accuracy and less maintenance.

To achieve this ideal, the sensor should be placed via ball valve insertion (hot tap), into a main process line at least 10 pipe diameters from any sources of turbulence such as pumps, valves, elbows or any device that could cause a sudden pressure drop in the process piping. Because of the density and high particulate content of the process fluid, the sensor should be inserted just into the process fluid such that it is in constant liquid contact with the fluid but is not in the full force flow of the stream (Fig. 13).

A horizontal run of pipe makes for the best sensor location. Although a TBI-Bailey sensor can be mounted in any orientation, the ideal mounting is between 1:00 and 5:00 (Fig. 14). In processes that have a high particulate content, it is best to install the sensor at horizontal levels or a little higher to slow down gravity deposition of the particulates.

TBI-Bailey’s type J or type 3 glass is ideally suited to sugar mill applications. Both glass types are chemically resistant and function well in high temperature environments. The J glass is especially suited to applications that tend to coat pH sensors. It is also advisable to use a wood liquid junction in all of the applications except possibly the carbonation.
portion of the process where the pH is in excess of 10.5 pH. Then, Teflon® is the appropriate material.

**Calibration and Validation**

Calibration and validation...isn’t this the same thing? Absolutely not! To calibrate is to “standardize by determining the deviation from a standard to ascertain the proper correction factors.” This has traditionally been accomplished using buffer solutions. However,
an instrument calibration is far more precise when it is accomplished using electronic means (a millivolt source) rather than standard solutions (buffers) simply because the inherent error in a standard solution is much greater than that in a precision electronic device. Don’t worry, you will get to use your buffers in the validation section.

Most pH instruments allow you to calibrate both the input and the output. Since the output is simply a straight line of millivolts versus milliamperes, and since the output depends on the integrity of the input, it is best to calibrate the input first. You will want to calibrate the input zero followed by the input span. The method to accomplish this varies from instrument to instrument so look at the operations manual for your instrument before proceeding. The input zero should be adjusted at the low value for the instrument. This is usually either zero pH (414 millivolts) or zero millivolts (seven pH). The span adjustment is made at the high value for the instrument - typically 14 pH (-414 millivolts).

The output calibration is now simply a matter of choosing your pH scale for your four to 20 milliampere output signal and then setting it up electronically. You may choose from a variety of (usually linear) scales. The standard setup is zero to 14 pH equal to four to 20 milliamperes, but you may set most instruments up with higher resolution by squeezing the output. This is accomplished by choosing a scale such as two to 12 pH equals four to 20 milliamperes, or four to 10 pH equals four to 20 milliamperes.

Note: This practice gives better resolution in the output, but it neither changes nor improves either the precision or the accuracy of the instrument.

To calibrate the output, simply feed the low signal to the instrument, adjust the output zero, then feed the high signal and adjust the output span. Your instrument is now calibrated. “What about the sensor?” you ask. Well, that is where the validation part comes into play.

Validation is “the determination of degree of validity of a measuring device.” Validation of your pH loop is simply a matter of sampling the process fluid with a portable measuring device. This procedure is known as a grab sample. “What about my buffers?” you ask. You must use your buffers to calibrate the portable pH sensor and transmitter. Since this sensor spends most of its life in a buffer waiting to sample a process fluid, a buffer solution is an ideal media for its calibration. So, step one is to buffer-calibrate your porta-
ble device. Step two is to take a grab sample of the process fluid and step three is to make a one point (zero) adjustment to the online pH instrument.

It is extremely important when performing a grab-sample validation to make sure you are comparing apples to apples. In other words, the conditions for both fluids should be as close to identical as possible. Since pH is a function of temperature, the sample should be obtained and measured at the operating temperature. If possible, pull and measure the sample immediately...do not walk it back to the lab. Also, try to pull your sample as close as possible to where the pH sensor is located in the process (10 feet away is sometimes too far away) to insure that chemical and physical changes have not occurred in the fluid that could change the solution pH. Your pH loop is now calibrated and process validated and is ready to accurately measure and possibly control your process pH.

You can learn more about calibration and validation techniques by referring to your pH instrument manual. More information is also contained in Bailey Technical Papers T-P90-2, Calibration and Troubleshooting of pH Loops and T-P93-11, Validation of Field pH sensors and Temperature, Another Wild Card in pH Control.

**Maintenance and Troubleshooting**

Your online pH sensor is going to require some cleaning from time to time. The better the installation, the less time you will spend cleaning the sensor.

Most of the applications in the sugar mill are at a neutral or slightly alkaline pH so typically, the best cleaning fluid is going to be something acidic. The usual procedure is to pull the sensor from the process, rinse it well with DI water (or tap water if DI is not available) and then soak it in a five to 10 percent HCl solution for five to 10 minutes. Rinse the sensor with copious amounts of water and return it to the process. Wait about 20 minutes for the sensor to come to chemical and thermal equilibrium. The sensor may bounce back within a minute or two, or it may take the full 20 minutes. The time depends on the temperature difference between the process fluid and the cleaning fluids used on the sensor and the amount of acid entrained in the liquid junction after cleaning. As soon as the process temperature is reached and the cleaner has dissipated from the liquid junction, the sensor can be considered accurate. At this juncture, you will probably want to revalidate the loop (see the previous section).

Troubleshooting should not pose too much of a problem since there are only two devices in the loop - the instrument and the sensor. If you suspect a problem with the instrument, try a millivolt calibration. It is helpful if your millivolt source has a high impedance switch on it so that you can make sure there is no damage to the front end of the instrument. Simply throw the switch while looking for a sudden change in the displayed pH. If there is no change, your electronics are probably fine.

The sensor can cause more of a headache in the troubleshooting department. First, visually inspect the sensor. Is the measuring electrode cracked, broken or completely missing? If so, it is time to replace the sensor. Is the bulb or liquid junction area visibly coated? If so, clean the sensor and perhaps shorten the normal interval between cleanings.

If the sensor appears intact and clean, then a buffer check is in order. Check the sensor in two buffer solutions to determine if it responds to changes in pH with alacrity and to see if it is short-spanning or has an unusual offset. It is best to use 4.01 and 7.00 NBS traceable buffers that are fairly fresh and have been sealed off from the environment. If the sensor does not respond to changes in pH when moved between the two buffers, then it probably has an internal electrical short. If the sensor wanders all over the scale no matter what you
put it in, it may be broken internally or the liquid junction may be completely plugged up. Try cleaning the sensor and then retest it. If it continues to malfunction, it will need to be replaced.

If the sensor is sluggish in its response to changes in pH or it short spans (less than 2.7 pH span between a four and seven buffer), it is either dirty or old. Try cleaning. If upon retesting, it is still sluggish or short spanned it is probably at the end of its useful life (all of the active sites on the glass have been depleted) and must be replaced.

Finally, if the sensor responds quickly and has a three pH span between the four and seven buffer, but has a large offset (measures nine in a seven buffer and six in a four buffer), there may be some problem in the reference area of the sensor.

The usual pH sensor problems that crop up in the sugar mill are breakage and coating. Breakage requires replacement and coating requires cleaning. Sometimes it is necessary to relocate the sensor to a cleaner location - possibly someplace with better flow.

Conclusion

Whether it is sugar beets or sugar cane, pH control in the sugar mill is a critical parameter in the separation of sucrose from the fiber of the plant being processed. It is also essential in facilitating the removal of contaminants that are formed in the various reactions such as the precipitation of calcium carbonate.

Sugar milling and refining operations have not changed significantly for many years. However, the measurement and control of pH in the mill has changed tremendously in the last 10 years primarily due to improvements in the durability and accuracy of the equipment. pH control is now achievable because there are devices available that will survive in the high temperature, harsh chemical environment of the sugar mill.

The keys to successful pH control are: Use a tough pH sensor. Put it in a smart location. Keep it clean so that it can do its job.

References
