Sugar News 1
Perspectives in Liquid Process Analytics

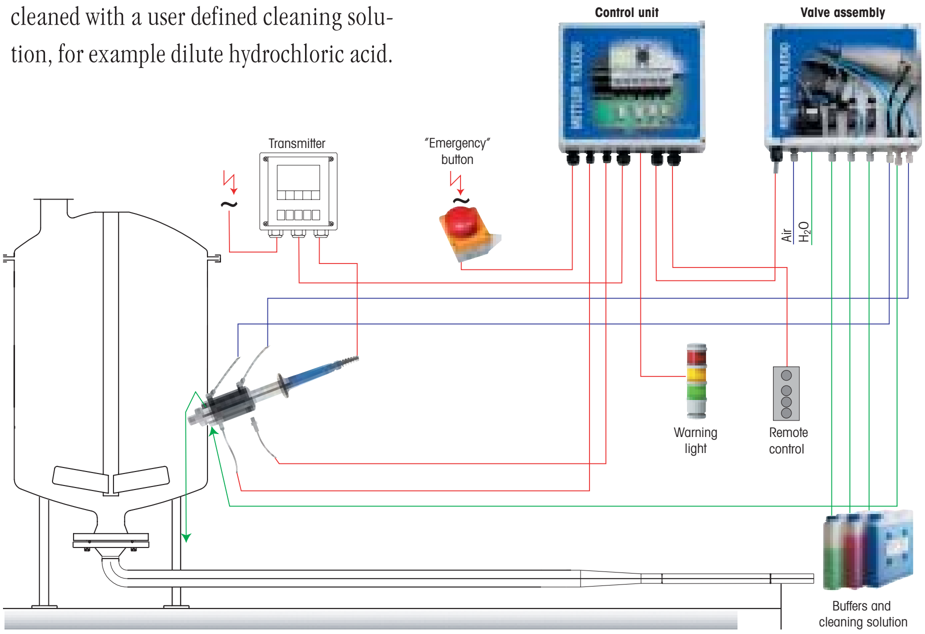
pH and DO sensor maintenance-made easy

Depending upon the type of application and actual operating conditions, sensors used in pH, redox and dissolved oxygen measurement may require more or less frequent maintenance attention. Particularly in processes which tend to produce a coating on the sensors, it is necessary to clean, test and calibrate the sensor at regular intervals.

Automation of these tasks can result in a substantial increase in measurement accuracy and service life of the sensor. By automating these tasks, the measurement accuracy and lifespan of the sensors can be decisively improved and the manpower requirement for maintenance work substantially reduced. The use of automatic cleaning systems leads to cost reductions within a short period of time.

The new generation of EasyClean systems also ensures the highest degree of process safety and reliability. Typical areas of application are to be found in the sugar and starch processing industries, as well as in wastewater treatment. In these processes, stubborn deposits often form on the sensor surface. Through use of the EasyClean 300, the sensor is automatically flushed with water, and can be automatically cleaned with a user defined cleaning solution, for example dilute hydrochloric acid.

Control system for Easy Clean 300.



METTLER TOLEDO

Contents

SugarNews 1

Impressum

Publisher
Mettler-Toledo GmbH
Process Analytics
Im Hackacker 15
CH-8902 Urdorf
Switzerland

Production
MarCom
CH-8902 Urdorf, Switzerland

Technical articles
Mettler-Toledo GmbH
Prozessanalytik
D-35396 Giessen, Germany

Illustrations
Archive MarCom
CH-8902 Urdorf, Switzerland
and
D-35396 Giessen, Germany

As required, the cleaning system can be operated manually or fully automatically. The fully automated basic functions of flushing and cleaning enable optimal utilization of the sensors and maximize their service life.

A system is made up of the following modules:

– an easy-to-install and operate EasyClean unit
– a transmitter, e.g. type pH 2100,
– a reliable and safe, pneu matically operated retractable housing, type InTrac 777SLP or InTrac 77SLI

Flushing and cleaning cycles are triggered by a programmable timer. The controls regulate all valve operations, as well as the feed and discharge of cleaning and flushing solutions. In conjunction with the high-quality and durable process electrodes from METTLER TOLEDO, the most demanding requirements of industry are optimally fulfilled. ■

NEW: InPro® 4800

The high-class combined pH/ temperature electrode

METTLER TOLEDO presents a new, leading class, combined pH-/temperature electrode. This sensor has been specifically designed for use under the most severe process conditions, particularly those found in the industrial and chemical processing industry. It offers reliable measurements in media which are either oxidizing or have high acid or alkali content. A special version is also available with a flat glass membrane and integrated potential equalization (solution ground-"SG") for media with a high proportion of fibers and particles.

Specification	InPro 4800
Measuring range	pH 0...14
Temperature range	5...130 °C, 23 ... 266 °F
Pressure range	0...13 bar/188 psi, triple safety
Reference system	pressure-compensated double-chamber system with gel electrolyte, Ag/AgCl lead-off
Diaphragm	external: PTFE circular diaphragm internal: special ceramic
Glass membrane	InPro 4800 cylindrical, membrane glass highly resistant to alkalis InPro 4801SG flat, low-resistance membrane glass (pH range 1...11)
Shaft material	glass shaft
Connector	VarioPin (VP), PG13.5 thread, protection rating IP68
Potential equalization	platinum auxiliary electrode (InPro 4801SG only)
Diameter	12 mm
Overall length	InPro 4800: 120, 225, 425 mm InPro 4801SG: 120 mm

Deviation between pH values in production and those from the laboratory: only an apparent difference

In sugar production, the pH value during the carbonation stage is an important measured variable for process control. Frequently, the online measured values are compared with parallel laboratory or mobile in-situ measurements. Deviations of up to 1 pH unit are common and require interpretation.

Features: InPro 4800

- Low maintenance electrode design – no need to top up electrolyte
- Prolonged useful life: very high resistance to oxidizing media that is strongly acid or alkaline, as well as to solvents
- High resistance to electrode poisons (e.g. sulfide) due to long diffusion path
- Correct measurement results owing to dirt-repellent PTFE circular diaphragm
- Can be used at particularly high process pressures because of its pressure compensating reference system
- Designed for use at high temperatures
- Integrated temperature probe
- Optionally supplied with flat membrane and integrated solution ground, particularly suitable for applications in processes with high particle or fiber content
- Reliable pH measurements over the entire pH range
- VarioPin (VP) connector, robust and waterproof (IP68):
- EEx certification: II 1/2G EEx ia IIC T6/T5/T4
- Certified in accordance with the new Pressure Equipment Directive 97/23/EEC, Art. 3, Para. 3, triple safety

Which measurements are correct? Those taken in production or those from the laboratory? Discussions frequently arise whenever the two values do not match. As a rule both measurements can be correct. The resulting differences can be explained by the influence of temperature on measurement.

Every medium alters its pH value relative to temperature

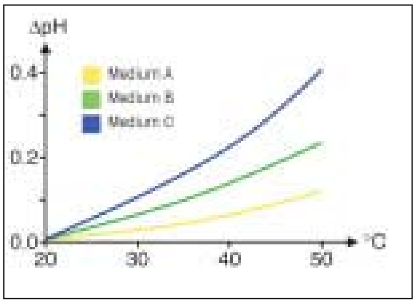
And indeed quite considerably. Generally, this phenomenon is not sufficiently known, and the measured value therefore is not compensated. This means that a sample measured at room temperature and one measured at a process temperature of 70 °C / 158 °F show different, deviating pH values. It is therefore imperative when measuring a pH value, to measure and document the temperature (of the sample medium) at exactly the same time. Moreover, if pH values are to be compared with each other, then the individual measurements must all be made at the same temperature. This has been clearly shown during sugar production. If the (grab) sample is heated to 70 °C / 158 °F in the laboratory and the pH value then measured, the value will concur with the value of the corresponding inline measurement.

Elimination of systematic deviations

The described deviation tendency is well reproducible at constant process conditions. The delta function of some transmitters allows the displayed value of the process measurement to be adapted to the laboratory measurement value. The delta function enables a fixed, freely programmable factor to be added to or subtracted from the displayed value. Measurement values from production or laboratory can thereby be brought into relation with each other.

Theory and practice of pH measurement

The measurement of pH values has many different aspects. METTLER TOLEDO makes relevant literature available for process operators, and offers technical seminars and workshops where the theory and practice of pH measurement is dealt with competently and comprehensively. Further information can be obtained directly from your local METTLER TOLEDO representative.



Temperature effects of different media.

pH control in sugar processing

White gold

from princes' condiment to peoples' commodity

Historians suggest that sugar was first systematically harvested as a food in Polynesia, maybe as long as 5,000 years ago, where it grew as sugar cane. It was discovered in India in the 5th century BC and is reputed to have been brought to the Mediterranean countries by Alexander the Great in the 4th century BC. Sugar subsequently made its appearance in England around 1100 AD, having been brought back by returning Crusaders.

Since sugar cane can only be grown in tropical and subtropical climates, for centuries it was a luxury, only affordable by kings and princes who could pay the price of importation from far away countries. Five kilos of sugar could buy a whole suit of armour for a knight in the Middle Ages! Furthermore, a one hundred ton shipload of sugar was valued at around $1,600,000 at today's prices. The "white gold" was purified in apothecaries shops and sold in small quantities as a medicine for the sick (so, it didn't always have to taste nasty as granny used to tell us).

User friendly programming at the touch of a button.

Because of the high price, many attempts were made to obtain sugar from other plants. Early experiments extracting sugar from maple syrup were soon abandoned because the yield was too small. It was not until 1747 that the German chemist Andreas Sigismund Marggraf discovered that the same sugar as that in sugar cane was present in beets. The following year he successfully found a way to extract the sugar and in 1799 the first beet sugar factory was built at Breslau. Thus the "white gold" became an affordable basic foodstuff and it is difficult to conceive a world without it today. Sugar is now produced in 127 countries worldwide, about 40% from beet.

Sugar and pH measurement

Sugar processed from cane sources comes as "raw sugar", 95-99% sucrose, and is ready for immediate refining. Beet sugar has first to be extracted. The preliminary cleaning of the beets can release small quantities of sugar into the 'flotation water'.

Microbiological processes (conversion of sugar into acids) can have an adverse effect on the quality of the "flotation water". To avoid acid corrosion of pipes, pH control ensures the maintenance of a defined pH,

Sugar production area

Delivery of sugar beet.

often by dosing with lime solution. After cleaning, beets are chopped and extracted with hot water (about 70 °C / 158 °F). The pH and hardness of the water are critical for this process. Hardness should be about 100 degrees DH and the pH should lie between 5.2 and 5.8, necessitating appropriate pH control.

From this point on, refining of beet or cane sugar is quite similar, differing only in exact temperatures and pH required. The refining process removes all non-sugar materials such as colour and flavour, producing white sugar, brown sugars and syrups. Most of the non-sugar materials end up in molasses, an important byproduct used in industrial fermentation and animal feeds, for example.

Critical pH control

An important stage in the purification is the addition of "milk of lime" (calcium oxide, or "quicklime" slurry), to bind the impurities and its subsequent precipitation as the carbonate, a process known as "carbonatation". The initial addition of "milk of lime" typically raises the pH to between 10 and 11, under controlled conditions, to provoke an effective precipitation. Carbon dioxide (often recovered from fuel combustion in other parts of the process) is introduced, keeping the pH above 10. The calcium carbonate (chalk) formed attracts colourants, resins and other impurities in the liquor, effectively removing them. This is the "first carbonation" and the filter cake from this stage (which still contains a fair amount of sugar) is washed to recycle the sugar back to an earlier stage in the process. The resultant cake is then sold to farmers as a soil conditioner, so nothing is wasted.

The liquor from the first carbonation still contains traces of calcium hydroxide. To remove these last traces, a second carbonation takes place at 90° - 95 °C (194 °/ 203 °F), with the pH usually ending somewhere between pH 7.5 and 9.0. After a further filtration, the liquor is ready for final processing by evaporation of water in vacuum pans to provide crystalline sugars and syrups.

Electrode contamination

In both carbonations, pH is a decisive parameter for the success of the operation. However, due to the high temperatures and the presence of high concentrations of lime, the measurement of pH is particularly difficult. Layers of lime often coat the whole electrode, junctions clog up after very little use, and sulphur compounds from the bleaching stages can react with silver ions escaping from the reference system of a pH sensor at the diaphragm, causing sluggish responses and inaccurate measurement. The high temperatures involved also militate against long sensor life.

A further complication is that refining 'campaigns' are non-stop affairs running 24 hours a day, 7 days a week.

Modern process control systems control production.

Latest developments

In these circumstances, automatic control of the pH sensor has proved particularly successful. METTLER TOLEDO automated systems such as the Easy Clean®system, coupled with liquid sensors filled with CALCOLYT (a lime dissolving electrolyte), and our patented "silver ion barrier" reference system to avoid escape of silver ions, have been installed worldwide.

Automated EasyClean systems.

The measurement chain can be automatically removed and cleaned at programmed intervals, increasing the confidence in and repeatability of the pH measurement and saving considerable overhead in manual maintenance. ■

Wastewater monitoring: efficiently and economically

METTLER TOLEDO offers a complete selection of measuring systems especially for monitoring industrial wastewater, and for wastewater treatment. They can be used to measure pH values, redox potential, electrolytic conductivity and oxygen content.

Value Line

Economical transmitters

These devices are easy to use and offer more than just basic functions, such as
– automatic calibration
– temperature compensation
– controller function
– 2 current outputs for signal processing
– 2 limit contacts
– alarm- and wash contacts

Mounting can be either directly into a control panel cutout or in an optional stand-alone enclosure, as desired.

Value Line transmitter for wastewater control and treatment.

It's the sensor that matters

INGOLD electrodes from METTLER TOLEDO are renowned for their long service life and reliability. For more than 40 years they have been used successfully in demanding biotechnology and chemistry applications. ValueLine sensors, which have been developed specifically for wastewater applications, offer many of the advantages of process sensors, at low cost.

The InPro 4010 pH electrode has a special polymer electrolyte that ensures its non-susceptibility to contamination even in heavily polluted wastewater. The InPro 6050 oxygen sensor needs very little maintenance, is easy to service and has a long service life. Owing to the polarographic principle of measurement, the sensor is virtually independent of the flow.

Numerous options are available for process adaption of the sensors: immersion, insertion and flow-through housings as well as retractable housings changeover. In conjunction with the EasyClean series of control systems, the sensors can be flushed, cleaned and calibrated automatically in-line where required. This reduces maintenance effort to a minimum, while measurement accuracy and service life of the sensors is optimized. ■

pH sensor InPro 4500
Oxygen sensor InPro 6050
pH electrode InPro 4010

Online oxygen measurement during yeast fermentation

Nowadays, yeast is generally produced on an industrial scale. The necessary biotechnological plant (bioreactors) must be fully suited to the physiological requirements of yeast metabolism in respect of substrate feed, aeration, temperature, and pH control. The basic substrate used in the production of yeast is molasses, a byproduct of sugar manufactured from cane and beets.

The influence of aeration

Optimal process utilization of the substrate is only possible if the supply of oxygen is adequate. Too rapid the addition of substrate (e.g. molasses) can lead to so-called catabolite repression and a "switch" to anaerobic fermentative metabolism, with an unintentional formation of ethanol. However, even correct dosing of molasses does not rule out such an effect, since inadequate supply of oxygen is the most sure and direct way to alcohol fermentation. What is a welcome factor in breweries is detrimental to yeast production, resulting in a substantial loss in yield.

Inadequate oxygen supply is also responsible for a higher level of pollution in the (fermentation) waste liquor, since during subsequent downstream separation processes, the effluent fraction contains a substantially higher proportion of (aerobically) non-degraded nutrients. "Over-aeration" of the fermenter would certainly be one way of ensuring an adequate supply of oxygen, on the other hand it would drive up energy costs considerably.

Continuous inline monitoring of the oxygen supply helps the plant operator to optimize the yeast fermentation process both relative to product yield and to effluent pollution load. Oxygen measurement systems from METTLER TOLEDO are able to provide important real time data concerning the oxygen supply situation. First, it is possible to take direct measurements of the oxygen concentration (dissolved oxygen) level in the fermentation broth, and second, the oxygen uptake rate can be calculated on the basis of measurement and comparison (differential) of the oxygen content in the feed air and in the exhaust gas.

Result

Through online oxygen measurement in yeast fermentation, yield rates can be optimized, effluent pollution load reduced, and energy costs saved. ■

The low-maintenance InPro 6800 - a new generation of dissolved oxygen sensors for fermentation processes.

Regular maintenance increases safety and avoids production failures

O-rings have the important task of sealing moving parts such as sensors and housings in order to prevent the escape of medium. Mechanical and chemical stress effects in everyday production are so great, that O-rings should be replaced at regular intervals.

Mechanical stress intensifies in relation to the increase in solids content and crystallinity of the (product) medium. The degree of chemical resistance depends upon the material composition of the O-ring, and is already commonly known for many chemicals. The most important specifications of the materials of the O-rings supplied by METTLER TOLEDO can be seen in the table.

Due to the chemical and mechanical strain placed on O-rings in industry, METTLER TOLEDO recommends that the O-rings are replaced at least once a year. It is advisable to carry out replacement work 4 to 8 weeks before the start of the campaign.

O-rings can be replaced on site or at METTLER TOLEDO Service Center. Qualified service technicians carry out replacement according to the manufacturers' instructions and test and verify the operation of each retractable housing. If O-ring replacement is performed at a METTLER TOLEDO Service Center, the retractable housing is also subjected to a pressure test.

O-Ring-Material	Characteristics
Viton®	Well suited for use with many chemical liquids, particularly combustible products and fuels; not suitable for use in hot CIP alkaline solutions; high thermal stability
EPDM	Well suited for CIP operations in acids and leaches; moderate thermal stability
Silikon	Well suited for fats and oils, low thermal stability
Kalrez®	Chemical resistance, rubber-like elasticity of elastomers; high thermal stability

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