



the food industry

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FI Europe 2003

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Brau Bevale

Glassless pH
analysis using
Isfet sensors



**Optimum texture using
starches which swell in cold water**

Plus **SPECIAL FEATURE** **HYGIENIC DESIGN**

No Glass

Online analysis of milk using ISFET Sensors

Matthias Klüh, Dipl. Ing.

Durafet II is a pH sensor suitable for use in industrial applications. Measurement is based on an Ion-Sensitive Field Effect Transistor device as an alternative to traditional glass style pH electrodes. The advantages of this sensor over glass pH electrodes are a faster response time even at low temperatures, greater robustness due to the fact that it contains no glass, and also negligible acid and base errors. With these characteristics, the sensor is evidently suited to use in milk processing.

In the manufacture of milk products, measurement of the pH value of the raw milk at the process intake is a key parameter. In this specific case, the milk is delivered every half an hour and pumped into large storage tanks. Previously, a “grab” sample has been taken from each delivery and tested in the laboratory to determine its pH value. Owing to the inevitable time lag between the sample being taken and

the results of the analysis being available, the whole quantity in storage must be discarded if the measured pH value lies outside the permitted acceptable limits.

Here the implementation of online pH measurement presents clear advantages. Online measurement makes it possible to validate the quality of incoming milk pH values prior to use in the process. Another benefit of direct

benefit of direct in line measurement is that the workload involved in carrying out pH analyses in the laboratory is reduced to a reasonable level. There are essentially two obstacles to online analysis in this application using conventional pH sensors. Since measurements are taken directly from the milk, the use of glass is out of the question – in the event of a sensor being defective or breaking, there would be too great a



Illus. 1: pH measurement point with Durafet II sensor in casing



Illus. 2: Sanitary pH sensor in casing

risk of the milk being contaminated by splinters of glass. The milk is also delivered at low temperatures at which conventional glass sensors have slower response times.

Advantages of the Durafet II ISFET sensor

These obstacles can be circumvented using Honeywell's Durafet II pH sensor. A semiconductor chip functions as the pH-sensitive measuring element - there is no need to use glass in the sensor. In addition to this unique design feature, the measurement system adopted has additional advantages that are due primarily - in comparison with conventional glass sensors - to the difference in chemical interactions between the sensor and the medium to be analysed.

These interactions can best be explained if one considers the measurement system used in an ISFET pH sensor. In conventional field effect transistors (FETs), the current between drain and source is controlled by the application of an applied voltage at the gate. In a FET intended for use in pH measurements, the gate is used as an electrically insulating, ion-selective film. This film is in direct contact with the medium to be analysed. With this arrangement, the medium itself acts as the gate. The FET is activated, and a varying electrical potential is generated based on the electrical potential of the media being analysed. In summary, the ISFET junction responds to pH of the media analogous to that of glass pH electrodes. As an example, if solution pH increases by one pH unit the potential at the gate / solution interface becomes more negative by approximately 60 millivolts.

With conventional sensors there is also the effect of the deterioration of the pH electrode glass envelope, often resulting in frequent pH sensor replacement. In high alkaline applications, errors caused by the interference of other Ions in the media (typically Sodium Ion) can also present a problem for the end user. The nature of technology used in ISFET pH measurement significantly reduces interference of this type. Marked changes in the temperature of the medium and the relatively low temperature of the milk do not adversely affect the accuracy of the

measurement, or the electrode response time.

Non-glass pH electrode technology facilitates direct on line / in line / in process pH measurement with very little risk of process contamination.

Hygienic design

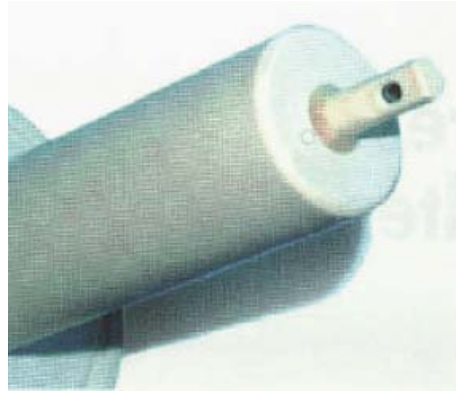
To meet hygiene requirements and to minimise changes to the existing process, the customer selected Honeywell's model 7794 Sanitary Durafet II pH sensors with a 1.5" diameter process flanges. These sensors are made of high-performance food-grade polysulphone plastic. The process flange forms an integral part of the electrode body. The body (whose surface roughness is less than 0.8 µm) is designed specifically to meet hygienic requirements to 3A standards.. Honeywell's unique method of sealing the ISFET measuring electrode and also the ceramic reference junction into the electrode body ensure that the integrity of the sensor maintained.

The only change implemented by the customer was to install T-pieces with 1.5" tri-clamp flanges on the existing stainless steel pipes that transport the milk. The sensors are placed directly in the stream of the medium, typically under pressures of approx. 6 bar. The sensor signal is analysed by the DirectLine DL 421 pH transmitter. The DL421 transmitter module enables a simple and cost – effective method of converting measured pH values to 4-20 milliamp signals for end user monitoring and control.

Operated with just three buttons

The DirectLine DL421 pH transmitter uses 2-wire technology together with the latest digital technology. The robust and watertight plastic casing with IP 66 protection can easily be installed in small areas owing to its compact size. The DL421's output is in the form of a 4.20 mA electrical signal, which is not vulnerable to interference. The parameters are set, and the module operated, using just three buttons mounted on the transmitter front face. Settings and operational status are indicated by an LCD display also mounted on the transmitter front face.

When configuring the unit, messages are displayed on the LCD indicating the status of the required operating parameter. Inbuilt software allows for



Illus. 3: The measurement surface on the Durafet II sanitary sensor

sensor problem diagnosis.

Integral buffer recognition values enable calibration to be carried out quickly and simply. After each successful calibration of the sensor, the display shows the offset and percentage theoretical slope of the sensor. This diagnostic information makes it easy to assess the integrity of the sensor performance.

The ease of calibration and the convenient user interface with only three buttons in particular, were decisive factors leading the user to choose this solution. For simplicity of calibration, the DirectLine pH transmitter is mounted directly beside the sensor using a din style remote mount bracket supplied with the unit. The transmitter is connected electrically (4-20 milliamps) to the customers control system by a loop-powered cable.

For the purposes of regular checking and calibration in this particular application, the sensor is taken out of the process and taken to a calibration station along with the attached sensor cable along with the DL 421 pH transmitter. This needs to be done because, given the confined spaces, the sensor cannot be inspected directly at the measurement point.

It may be that in other applications at this customer site, the conventional method of on-the-spot calibration and cleaning will be carried out.

The Model 7794 pH electrode remains in the pipeline when process cleaning is carried out.

Because of this, consideration needs to be given to the electrodes resistance to highly acid or alkaline cleaning products at relatively high temperatures. CIP (clean in place) cleaning puts the greatest wear and tear on pH sensors, since typical CIP solutions at temperatures around 85° C not only corrode conventional glass electrodes but also markedly affect the lifetime of ISFET pH sensors.

There are two ways to achieve acceptable sensor lifetimes. On the one hand, the sensor can be taken out of the process during cleaning, by means of a manual or automatic device. This means however, that the sensor is not cleaned together with the process – a separate cleaning procedure needs to be carried out. The second option is to make the Semiconductor chip even more resistant –considerably so– to chemicals such as NaOH. This is a more technically demanding approach.

Positive experience in day-to-day operation

Online pH analysis has been in use with this customer for over one year. The user's expectations have been confirmed by their experiences gained over this period. The sensor responds quickly to changes in the process pH and shows good values for electrode slope and zero point. The ease with

which the Model 7794 Durafet II sanitary pH can be installed and removed combined with easy-to-use DirectLine DL421 with its integral display and function keys, won over the customer.



Illus. 4: DirectLine DL421 pH value measurement converter

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pH Applications

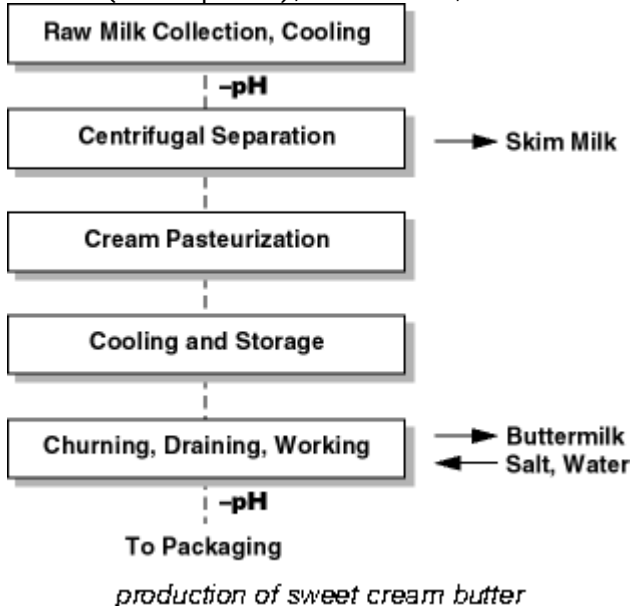
pH Measurement in Butter Making

New Non-glass pH Electrode Capabilities

- Reliable Durafet® solid state pH electrode is designed for dairy sanitary service.
- Authorized to use 3-A symbol
- Ion-Sensitive Field Effect Transistor (ISFET) sensor eliminates fragile glass membrane
- Fits standard tri-clamp or equivalent flanges
- Fast, 1-second response is typical
- Measurement is accurate, with no sodium or ORP interference.

Background

Buttermaking is the process of converting (churning) cream, an emulsion of butterfat-in-serum (water phase), into butter, an emulsion of serum in butterfat.



Typically the cream contains about 40% butterfat and the finished butter contains about 80%. Other ingredients include water, non-fat milk solids (curd) and salt. In Europe, the butterfat content is slightly higher and no salt is added. pH measurement can be significant in the process.

The steps of buttermaking are outlined in the accompanying figures. The pH of raw milk is typically between 6.5 and 6.8. pH measurement can be used to detect the presence of lactic acid which developed by microbial action, which lowers the pH and affects the flavor. In some cases depressed pH in milk or cream can be corrected by neutralizing with sodium carbonate or sodium hydroxide. The raw milk is centrifugally separated into cream and skim milk. Next the cream is pasteurized using either a batch heat

treatment or the more prevalent continuous HTST (high temperature, short time) process.

The cream is then cooled and usually stored overnight before churning. A few batch type churns are still used but most butter is made with continuous churns which rapidly agglomerate the butterfat into popcorn size granules. A byproduct is buttermilk, which is drained off. Then, the desired amounts of water and salt are worked in. In large modern plants, the finished butter goes directly to packaging machines for consumer size packages. It has been found that butter has an optimum flavor at a final pH near 5.

For the production of cultured cream butter, which is preferred in some areas of the world, appropriate bacteria culture is added to the cream following pasteurization. This culturing or controlled cream ripening process can be accurately monitored with pH measurement as the acid develops.

pH Electrode Requirements

pH electrodes must meet sanitary requirements for direct contact with food. The non-glass, Durafet electrode is a major breakthrough for pH measurement in this type of application. Based on new ISFET (ion-sensitive field effect transistor) technology, it eliminates the fragile glass membrane, the associated risk of breakage and liabilities of glass contamination of product and costly downtime. The electrode design complies with 3-A Sanitary Standards and is authorized to use the 3-A symbol.

In addition, Durafet electrode response eliminates the high impedance circuitry and vulnerability to insulation breakdown of glass membrane electrode signals. They have no ORP (oxidation-reduction potential) interference and negligible sodium ion error at high pH. They have exceptionally fast pH response—typically within 1 second. This assures close monitoring and control.

Long life of the solid state pH sensor is complemented by a reference electrode section with large surface area junction and gel electrolyte. The large junction area resists clogging and eases cleaning. No pressurization of the reference electrode is required. The probe also includes a temperature compensator to provide a compensated pH signal compatible with a variety of instrumentation.

Durafet electrodes can be used with the full line of Honeywell pH analyzers and two-wire transmitters. In addition, adapter modules are available to electronically interface the Durafet electrode to most other types of analyzers.

A simple solution.

Tomorrow's standards that Honeywell is setting today with the Durafet pH electrode include:

- Solid-state ISFET sensor is virtually unbreakable.
- Response has no sodium ion error or ORP interference.
- Flush reference junction and gel electrolyte reduce maintenance costs and increase electrode life.
- Built-in counter electrode improves measurement stability.
- Easily retrofits to existing systems, with sanitary flange and choice of insertion depth.

The sanitary Durafet pH electrode is another example of Honeywell's technology giving you a simple solution that increases productivity, ensures product quality, saves time, and increases process profitability.

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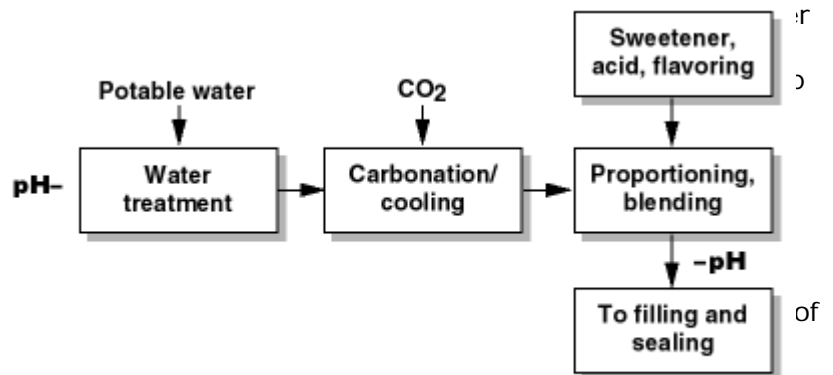
pH Applications

Measurements in Soft Drink and Juice Processing: Continuous Monitoring Essential to Product Uniformity

pH Measurements in Soft Drink and Juice Processing Water Treatment

Background

Water quality requirements for soft drinks are more stringent than for typical drinking water supplies. The seasonal variations found in many potable water supplies cannot be tolerated for a bottled beverage. The appearance and taste must be of consistently high quality after mixing with the other ingredients, and the beverage must have very good stability over a long shelf life.



soft drink processing

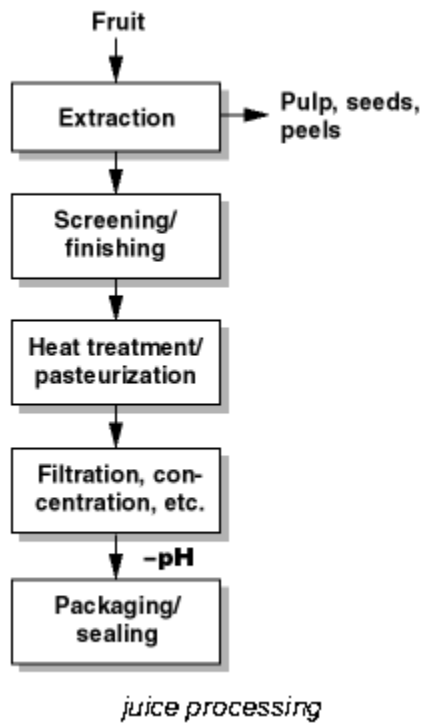
additional steps.

These may include: chemical addition (typically lime and alum) for thorough coagulation and flocculation, clarification, disinfection, sand filtration, activated carbon, fine filtration, and reverse osmosis. pH control is used to optimize the initial chemical additions in water treatment and also for the pretreatment for reverse osmosis membranes.

Acidity and pH of beverages

Acid in soft drinks imparts several desirable qualities. To the flavor, it adds tartness or sourness, modifies the sweetness of sugar and enhances thirst-quenching characteristics. Citric acid is widely used to sharpen the fruitiness of ginger ale and fruit flavored drinks. Phosphoric acid provides the flat sourness of colas, root beers and other nonfruit drinks.

As a mild preservative, acid extends the beverage shelf life. Some acids have more specific purposes. Ascorbic acid is frequently used in fruit juices as an anti-oxidant to preserve flavor/color. The preservative effectiveness of benzoic acid, a very weak acid itself, is enhanced by the lower pH developed by the stronger acids.



In juice processing, the contents of the fruit are extracted by squeezing or crushing. The pulp, seeds and peels are removed with screens in a finishing operation. The juice is then heat treated, filtered/concentrated as required by the particular product.

Several factors can affect acidity—fruit varieties, fertilizers, growing season conditions and ripeness—so monitoring pH is essential to control product uniformity.

Thus, the on-line measurement of pH provides continuous monitoring of free acidity, one of the important characteristics of soft drink and juice products.

pH electrode requirements

pH electrodes must meet sanitary requirements for direct contact with food. The non-glass, Durafet® electrode is a major breakthrough in the availability of pH measurement in this type of application.

Based on new, Ion-sensitive, Field-effect Transistor (ISFET) technology, it eliminates the fragile glass membrane, the associated risk of breakage and liabilities of glass

contamination of product and costly downtime.

The [Sanitary Durafet electrode](#) design complies with 3-A sanitary standards and is authorized to use the 3-A symbol.

In addition, the Durafet electrode signal level eliminates the high impedance circuitry and vulnerability to insulation breakdown of glass membrane electrode signals. They have no oxidation-reduction potential (ORP) interference and negligible sodium ion error at high pH. They have exceptionally fast pH response—typically within 1 second. This assures close monitoring and control.

Long life of the solid state pH sensor is complemented by a reference electrode section with flush junction and gel electrolyte. The flush junction resists clogging and eases cleaning. No pressurization of the reference electrode is required. The probe also includes an integral temperature compensator to provide a compensated pH signal compatible with a variety of instrumentation.

Sanitary Durafet electrodes can be used with the full line of Honeywell analyzers and two-wire transmitters. In addition, adapter modules are available to electronically interface the Durafet electrode to existing analyzers of nearly any manufacturer.

Tomorrow's standards that Honeywell is setting today with the Durafet pH electrode include:

- The solid-state ISFET sensor is virtually unbreakable.
- Response has no sodium ion error nor ORP interference.
- Flush reference junction and gel electrolyte reduce maintenance costs and increase electrode life.

- Built-in counter electrode improves measurement stability.
- The electrode easily retrofits to existing systems with sanitary flange and choice of insertion depth.

The Durafet electrode is another example of Honeywell's powerful technology, giving you a simple solution that increases productivity, ensures product quality, saves time and increases process profitability.

Recommended equipment:

- [7794 Sanitary Durafet pH electrode mounting](#)
- [9782 pH Analyzer/Controller](#)

[\[Return to Top\]](#) [\[Return to Applications Homepage\]](#)

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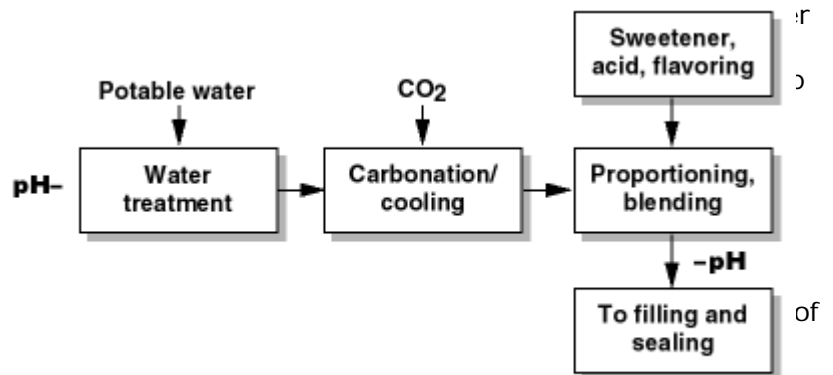
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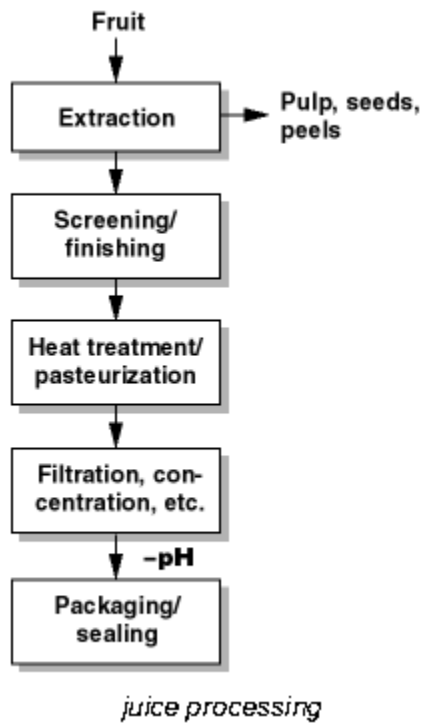
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Sanitary Durafet electrodes can be used with the full line of Honeywell analyzers and two-wire transmitters. In addition, adapter modules are available to electronically interface the Durafet electrode to existing analyzers of nearly any manufacturer.

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- The solid-state ISFET sensor is virtually unbreakable.
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- Built-in counter electrode improves measurement stability.
- The electrode easily retrofits to existing systems with sanitary flange and choice of insertion depth.

The Durafet electrode is another example of Honeywell's powerful technology, giving you a simple solution that increases productivity, ensures product quality, saves time and increases process profitability.

Recommended equipment:

- [7794 Sanitary Durafet pH electrode mounting](#)
- [9782 pH Analyzer/Controller](#)

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pH Applications

Cheese Processing pH Measurements

Accurate measurements ensure that your best ingredients produce the best products.

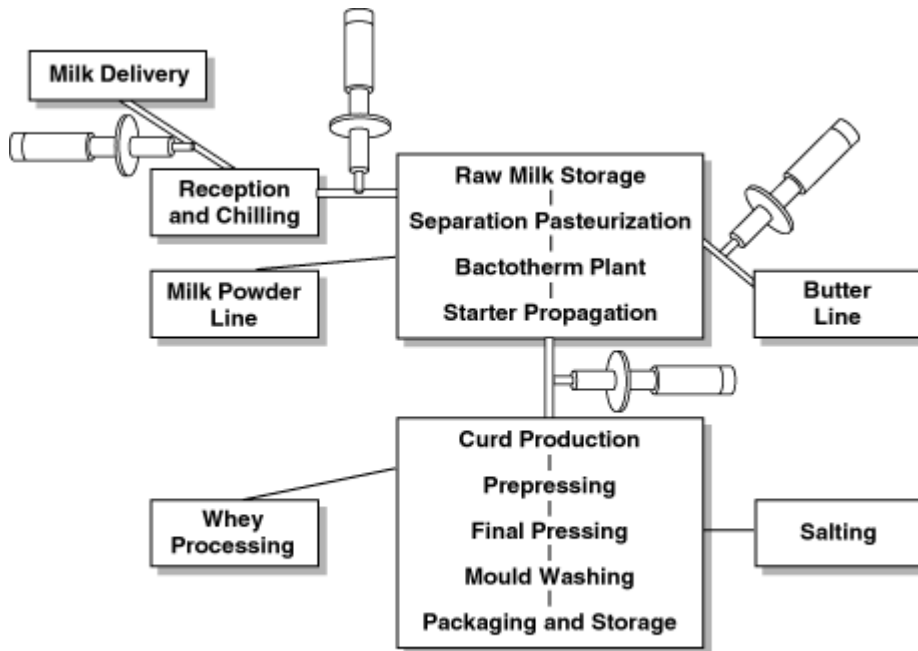
Background

Engineers need to know the physical properties of the product so that they can design the appropriate equipment and process. Operators must have accurate and reliable physical property data in order to ensure the quality of the product and the efficiency of the process. Reliable and accurate data is important in any production, but in food processing, the measurements also need to be from sanitary instrumentation. Here's one example of how Sanitary Durafet® pH electrodes can improve your on-line measurements and ensure your process's quality.

The process

Traditionally, cheese makers have developed a "feel" for time-temperature- acidity relationships for different types of cheeses. This "feel" is based upon certain criteria for acidity, curd development, heating times, and other process conditions. However, automation has led to certain equipment design principles and measurement techniques to ensure the "feel" of cheese production. These principles include:

- Production methods must produce cheese reliably and cost effectively, and with repeatability from batch to batch.
- Design should be simple and include an effective clean-in-place (CIP) system.
- Equipment must satisfy regulations.
- Design should permit installation in existing plants and be available with manual to fully automatic control schemes.



where the Durafet pH electrode helps your process the most

A cheese vat is a jacketed vessel equipped with hot and cold water supply for heating and cooling. The vat must heat the ingredients uniformly and at a slow, even rate during cooking. The setting temperature of the milk is dependent upon the starter bacteria and the type of cheese produced. The setting temperatures are usually in the range of 21° to 29°C (70° to 85°F). The final cooking temperature ranges from 40° to 57°C (104° to 135°F). During the cooking period the acid-forming bacteria will produce the desired levels of acidity before commencing with the next step of the cheesemaking process. Accurate pH readings can pinpoint the optimum time to cut the curd to achieve the highest yield of cheese and minimize the loss of solids into the whey. It is very important to have a reliable temperature and pH measurement to ensure the quality of each particular cheese. A cheese's distinctive flavor is determined in part by the pH balance of the process.

Flexible plant for multiple products

In today's fast paced manufacturing environment, how fast you can change your processing to meet your customers' demands often defines your margin of profit. In-line pH sensors can provide current information for controlling the process to yield consistently high quality product.

Advanced in-line process control.

Off-line sampling is slow, unresponsive, cumbersome, and costly. Now you can do in-line pH analysis with Honeywell's Durafet pH electrodes. The solid-state ISFET sensor is virtually unbreakable and is designed for sanitary service. Sanitary Durafet electrodes are accurate in a wide pH range (0-14), providing reliable measurements throughout the pasteurization process.

In-line sampling and on-line analysis enables you to correct process drift and protect product quality. The in-line Sanitary Durafet electrodes increase productivity, ensure product quality, save time, and increase process profitability.

Now you can do pH analysis on-line without disrupting your process for time-consuming pH lab analysis.

Durafet electrodes remain accurate under the most demanding conditions over a wide range of temperatures (-10 to +110°C). Sanitary Durafet pH electrodes feature a fast-responding temperature sensor for better control of food and dairy processes.

Reliable Sanitary Durafet pH electrodes provide a simple solution to a complex technology challenge.

Durafet pH electrodes are a series of rugged pH electrodes featuring a fast responding ion-sensitive field-effect transistor (ISFET) that provides increased system accuracy, stability, and reliability. The Sanitary Durafet pH electrode can be mounted in-line to provide on-line pH measurements. This increases your productivity, speeds control, improves product quality, and lowers installed costs.

The traditional glass membrane is fragile and delicate. When it breaks, it is costly and time consuming to replace. A solid-state sensor makes Durafet electrodes virtually unbreakable. Longer electrode life not only adds up to lower maintenance costs, but also provides increased reliability for even the most critical processes.

Six models of the Sanitary Durafet pH electrode range from 1/2", 2", and 3" flange sizes that have either deep or shallow penetration.

A simple solution

Tomorrow's standards, that Honeywell is setting today, include:

- **Solid-state ISFET sensor is virtually unbreakable.**
- **No sodium iron error or ORP interference.**
- **Built-in counter electrode increasing measurement stability.**
- **Designed for sanitary service and authorized to use the 3A symbol.**
- **Easy retrofits to existing systems, because it works with almost any analyzer.**
- **Complete selection of housings for in-line or immersion mounting.**

The in-line Sanitary Durafet electrode is another example of Honeywell's powerful technology giving you a simple solution that increases productivity, ensures product quality, saves time, and increases process profitability.

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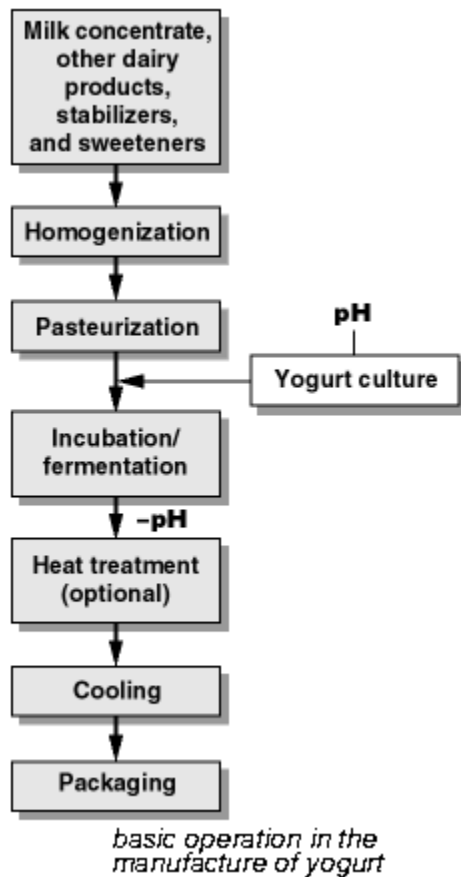
pH Applications

pH Measurement in the Manufacture of Yogurt: Ensuring Extraordinary Product Quality

Background

Yogurt is a popular, cultured dairy product produced by concentrated milk fermentation. The controlled production of lactic acid by fermentation is critical to the character of the product.

Lactic acid is responsible for the tart flavor and for destabilization of milk protein to form a gel structure. pH is an important measurement to monitor lactic acid production as well as for quality control of initial ingredients.



Yogurt production begins with the careful selection and blending of ingredients—such as milk concentrate, other dairy products, thickening agents, sweeteners and fruit—to give the proper solids and fat content, viscosity and flavor. The mix is homogenized at high pressure to prevent fat separation and assure solids dispersion. Heat treatment/pasteurization raises the temperature to 85-90°C, sufficient to destroy undesirable microorganisms and restructure the protein to improve viscosity. After cooling to 40-45°C, the sterile mix is ready for inoculation with smarter culture containing the specific lactic fermentation bacteria. Incubation can proceed either in the vat or in the package cups and take from 4 to 11 hours.

Fermentation converts lactose (milk sugar) to lactic acid, causing a pH drop into the range of 4.25-4.5. Bacterial action is then stopped by rapid cooling at the correct lactic acid level. The most readily available measurement for confirming the completion of fermentation is pH. Improper pH can be a cause of discoloration, excess free whey and excess or inadequate tartness.

pH electrode requirements

pH electrodes must meet sanitary requirements for direct contact with food. The [non-glass Durafet®](#) electrode is a major breakthrough in the availability of pH measurement for this type of application. The Durafet non-glass sanitary pH electrode now allows continuous, real-time monitoring

to accurately control fermentation.

Based on new, Ion-sensitive Field Effect Transistor (ISFET) technology, the Durafet electrode eliminates the fragile glass membrane and the associated risk of breakage and liabilities of glass contamination of product and costly downtime.

The electrode design complies with 3-A sanitary standards and is authorized to display the 3-A symbol. In addition, the Durafet electrode response eliminates the high impedance circuitry

and vulnerability to insulation breakdown of glass membrane electrode signals. They have no ORP interference and negligible sodium ion error at high pH. They have exceptionally fast pH response— typically within 1 second. This assures close monitoring and control of the end-of-fermentation point.

Long life of the solid-state pH sensor is complemented by a reference electrode section with large surface area junction and gel electrolyte. The large junction area resists clogging and eases cleaning. No pressurization of the reference electrode is required. The probe also includes an integral temperature compensator to provide a compensated pH signal compatible with a variety of instrumentation.

Durafet electrodes can be used with the full line of [Honeywell analyzers and two-wire transmitters](#). In addition, adapter modules are available to electronically interface the Durafet electrode to existing analyzers of nearly any manufacturer.

Simple solution

Tomorrow's standards that Honeywell is setting today with the Durafet pH electrode include:

- The solid-state ISFET sensor is virtually unbreakable.
- Response has no sodium ion error nor ORP interference.
- Large reference junction and gel electrolyte reduce maintenance costs and increase electrode life.
- Built-in counter electrode improves measurement stability.
- The electrode easily retrofits to existing systems with sanitary flange and choice of insertion depth.

The sanitary Durafet electrode is another example of powerful Honeywell technology, giving you a simple solution that increases productivity, ensures product quality, saves time and increases process profitability.

Recommended equipment:

- [7794 Sanitary Durafet pH electrode mounting](#)
- [9782 pH Analyzer/Controller](#)