pH Application Note

pH Measurement of Canned Foods

InLab Solids Pro-ISM Sensor

The code of federal regulations under 21 CFR part 114 requires acidic canned solid foods to maintain an equilibrium pH of 4.6 or below. To comply with the guidelines, it is imperative to frequently test and record the pH value of these foods during the manufacturing and production process. The use of state-of-the-art designed pH sensors with multifunction features, like METTLER TOLEDO's InLab Solids Pro-ISM, ensures accurate and precise pH measurement. This sensor enables piercing through solid sample mass to measure the pH of canned food directly, enabling faster analysis. Unlike a conventional pH sensor, this sensor has an open, and therefore clog-free junction. This application note discusses in detail the requirements for pH measurement of canned solid foods under 21CFR part 114 regulation and the features of this specialist sensor that helps to meet this requirement.

METTLER TOLEDO

Introduction

Canned foods grew in demand during the war period of 19th century among the military, and later went on to become an essential household requirement in Europe and America. They were specially famous for the preservation technique that made it possible to easily transport and store inexpensive and varied quality of high calorie, seasonal food products. However, to protect the food from serious health risks caused by the pathogen Clostridium botulinum, and to tackle other safety concerns, 21 CFR parts (Code of federal regulations) contains guidelines to control the canned foods industry.

A tin of canned food contains solid or semisolid food mixed with brine of known acid concentration. Normally, canned foods fall under two categories, acid foods and low acid foods. Acid foods have a natural pH of 4.6 or below whereas low acid foods includes foods other than alcoholic beverages having equilibrium pH greater than 4.6 and a water activity greater than 0.85. Typical examples of low acid food are meat, seafood, and vegetables. The low acid foods are acidified by adding acid or acid foods so as to maintain an equilibrium pH below 4.6. Every low acid food need not be acidified to maintain an equilibrium pH of 4.6 or below, and in that case, can be thermally processed as a low-acid food as per 21 CFR part 113 regulation. The thermal process makes use of heat to destroy or inactivate the potential microbes in the food sample. Alternatively, a competent processing authority should approve the reprocessing of the food and ensure it is a safe product. The person supervising the production of pH controlled acidified food products should be FDA approved as specified in 21 CFR 114.10 and should ensure compliance during the manufacturing process.

Importance of Measurement

As per the processes mentioned under 21 CFR 114.80, the required pH has to be achieved within the designated time in the scheduled process. These pH controlled foods need to be monitored and frequently tested at intermittent stages of the process for compliance. Potentiometric method of pH analysis are mandatory for food products having a pH above 4.0. Leakage or underprocessing of canned food causes growth of microbes, leading to spoilage. pH evaluation can help in screening the foods for presence of certain microbes, as listed in examination of canned foods chapter of Bacteriological analytical manual.

Measurement Challenges

For measuring the equilibrium pH of the final product, the accurate pH determination of the solid or semi-solid food with the brine drained off, is required. Direct insertion of the pH sensor into such food samples is difficult with the standard pH sensor. Moreover, interaction of the sample with the reference electrolyte can be restricted in a ceramic junction type sensor, due to the possibility of junction clogging. This effects the overall efficacy and accuracy of pH measurement, and the user may experience unstable and erroneous results.

The given table outlines the challenges and negative effects on pH measurement results of canned food samples when using a standard pH sensor.

Sample Challenge	Sample Impact
Solid or semisolid sample nature of the food sample	Direct insertion into such samples is difficult with standard shaped pH sensor.
Sample preparation	Alternate method requires blending the sample into paste for measurement and is time consuming.
High protein sample content	Sensor junction can get damaged due to clogging and precipitation causes inaccurate results.
High fat sample content	Insufficient miscibility of aqueous reference electrolyte with sample, which results in high response time.

Conventional sensors are inefficient at piercing through the sample matrix and the sensing membrane may get scratches during the measurement process. An indirect method for pH measurement of food samples require blending of the sample into a slurry to facilitate proper sample interaction with the sensor. As specified in 21 CFR part 114.90, the sample can be mixed with 10 to 20 ml of deionized water for each 100 grams of product. The pH analysis can then proceed as in a liquid sample. This method is time consuming and vulnerable to increased scope of errors during sample preparation.

Additionally, in the case of a conventional pH sensor with refillable electrolyte, high fat content in food samples makes it difficult for the outflowing reference electrolyte to mix readily with the sample components and attain a stable signal. Also, high fat and protein content accumulates on the sensor membrane, thereby leading to high response time and inaccurate results. Proteins from the food sample clogs a typical ceramic fritted junction due to precipitation, preventing electrolyte from further mixing with the sample and thereby causing measurement error.

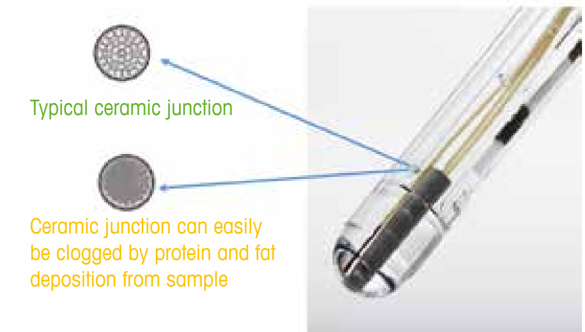

Figure 1: Sample impact on a conventional pH sensor having a ceramic junction

InLab Solids Pro-ISM for Reliable pH

The InLab Solids Pro-ISM (51344155) is a specialist sensor for measuring the pH of canned foods with accuracy and precision. The sensor is well designed to handle all the requirements needed for the pH measurement of canned foods. It has a built-in temperature probe and provides Intelligent Sensor Management (ISM) technology, allowing users to accurately capture all critical measurement parameters.

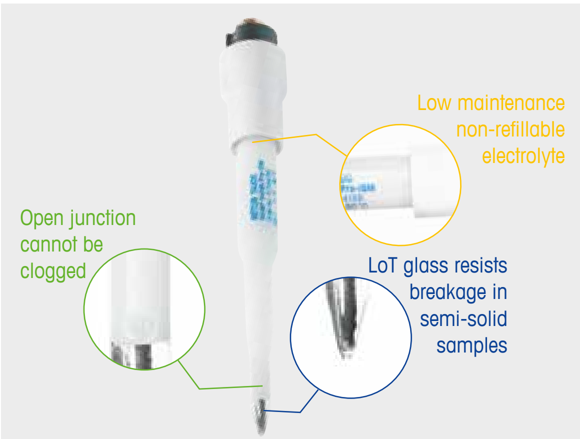

Figure 2: InLab Solids Pro-ISM pH sensor

The pH sensing membrane of this sensor is made of low temperature (LoT) glass, which has a low resistance and yields fast results. The sensor has a spear shaped tip made of toughened glass that allows direct insertion into the solid or semisolid food samples and is resistant to breakage. The low maintenance solid XEROLYT®EXTRA polymer reference system offers two benefits: it has a clog-free open junction which eliminates the risk of protein fouling; and the sensor interacts with the sample through diffusing ions, eliminating the difficulties associated with immiscibility of aqueous reference electrolyte with the sample. The specialized design and overall sensor technology of the InLab Solids Pro-ISM ensures direct sample measurement of any sample nature of canned food samples including solid food and liquid brine. This is critical for ensuring reliability and consistency in the manufacturing and quality control of such food samples.

Procedure and Method

The section 114.90 of 21 CFR discusses in detail the determination of pH by potentiometric technique and the required sample preparation of canned foods. The method requires to measure the pH at a temperature between 20 to 30 °C, optimum being 25 °C and recommends to use an automatic temperature compensation. METTLER TOLEDO pH meters and electrodes comply and are well above the required accuracy and other technical specifications, that are mentioned in the method. Standardization of the instrument and calibrating of the electrode with standard buffers are required by the method.

While performing the pH measurement, start with calibrating the pH electrode using buffers that bracket the sample range (in this case pH 4.01 and 7.00). Record the calibration slope and offset value for the electrode. A slope value in the range of 95 -105 % with a corresponding offset of 0 ± 30 mV ensures reliable pH measurement. The measurement should be carried out at an ambient temperature of 25 °C.

A typical canned food consists of a mixture of liquid brine and solid food. Normally, pH of brine is in equilibrium with the solid food if the acidification is properly done. 114.90 of 21 CFR recommends segregating the solid and liquid separately as per their solid to liquid packed ratio and analyzing the pH separately. If the liquid part from the brine or sample contains oil, it has to be extracted with water in a separating funnel and the pH of aqueous layer needs to be measured.

While using InLab Solids Pro-ISM pH sensor to measure the equilibrium pH of the solid, directly insert the electrode tip into the food sample Repeat the measurement at various positions to obtain a representative pH reading. Record a minimum of two pH values of the food sample. The sample is considered homogenous if the two readings are within ±0.05 pH units. The sensor can be used to measure the pH of brine too. Ideally, in an equilibrium condition, the pH value of solid food matches with the pH value of its brine solution. Proper care and handling of the sensor helps to maintain the sensor performance and optimum response time during the pH analysis.

Figure 3: InLab Solids Pro-ISM sensor to measure the pH Canned Foods

Results

Typical measurement results for acidified canned food items were recorded (in triplicates) using sensor InLab Solids Pro-ISM and are enlisted in below table.

Sample	Mean pH Value	Std.Dev.	Avg. time (s)
Pineapple slices	3.54	0.01	08
Halves Peach	3.67	0.01	06
Plum Tomatoes	4.22	0.01	08

Expert Tips

- For thorough cleaning of the sticky residues from the electrode surface after the sample measurement, clean it with Ethanol or acetone and then rinse with de-ionized water.
- Regular maintenance is very important for prolonging the lifetime of pH electrode. Periodic reconditioning of the electrode in 0.01M HCl is recommended, based on the sensor performance. Frequency of reconditioning would depend upon the number of samples analyzed per day and life of the sensor. An old sensor requires frequent conditioning compared to a new sensor. Remember to re-calibrate the sensor after reconditioning.
- Soaking the sensor in pepsin/HCl solution (51350100) for one hour once a week helps to remove protein build up on the glass membrane. Recommended frequency is once a fortnight or depending on the sensor performance. Sluggish response or inaccurate results indicate contamination of the glass membrane.
- The pH range for this sensor is 1 to 11 pH units and hence should not be exposed to harsh acidic (below pH 1.00) or alkaline (above pH 11.00) solutions.
- In between measurements or when the electrode is not being used for brief period, it is best to keep the electrode in wetting cap filled with InLab Storage Solution (30111142).
- Never store the electrode dry or in distilled water, as this will affect the pH-sensitive glass membrane and thus shorten the lifetime of the electrode.
- Ensure use of correct buffers in the correct sequence. Always use fresh buffers. Check the expiry date.

Further Information

- Electrode handling movies on:

YouTube

- Comprehensive range of pH meters, electrodes, solutions, and accessories:
  ▶ **www.mt.com/pH**

References

- Code of Federal Regulations. 21, Chapter 1 (4-1-04 ed.), part 114—acidified foods. U.S. Printing Office, Washington, D.C.
- Landry, W.L., Schwab, A.H. and Lancette, G.A., 1998. Examination of canned foods. Food and Drug Administration bacteriological analytical manual. AOAC International, Gaithersburg, MD, USA.

Mettler-Toledo GmbH, Analytical
Im Langacher 44
8606 Greifensee, Switzerland
Tel. +41 22 567 53 22 / Fax +41 22 567 53 23

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