Oxygen Monitors Optimize Quality and Flavor PART 2: Measure, Calibrate, Maintain

Introduction

Without monitoring oxygen levels throughout the brewing process, the finest hops, grain, and water won't add up to much. Oxygen is a tricky element to control. But, with the proper measuring equipment, oxygen can be kept in check, allowing brewers to ensure their product quality and increase batch longevity.

Benefitting from over 40 years of experience in oxygen measurement for the brewing industry, Hach (working with the Orbisphere brand) is well placed to evaluate both amperometric and optical technologies. This two-part application series examines the importance of monitoring oxygen using amperometric and/or optical dissolved oxygen (DO) sensors.[1,2]

PART 1 of this series covers critical topics when choosing an oxygen sensor, including:

- The effects of oxidation on the brewing process
- Amperometric and optical oxygen sensors
- Process conditions affecting oxygen measurement.

PART 2, in addition to reviewing background information on oxidation from Part One, covers information critical to measurement accuracy and day-to-day operations, including:

- True zero for oxygen sensors
- Sensor calibration
- Drift and sensor stability
- Response time
- Sensor maintenance.

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Accuracy of the Zero

As illustrated in Figure 1, the amperometric method can intrinsically provide a true physical zero (i.e. no oxygen means no signal). Whereas most amperometric systems show a drift of the zero and require a regular "zero" calibration, Hach's unique Orbisphere design guarantees a stable true zero over time. Laboratory and practical experience show that an accuracy as good as ± 0.1 ppb can be obtained with these amperometric sensors. Conversely, the parameter that varies the most with optical technology is the value in the absence of oxygen.

A zero calibration is generally performed by exposing the sensor to an oxygen free gas such as 99.999% nitrogen (N_2) or 99.999% carbon dioxide (CO_2). The accuracy of the zero is directly linked to the precision of the zero calibration, which is influenced by: the quality of the calibration sample (± 0.4 ppb), the absence of leakage in the calibration set-up, and the quality of the sensor signal. The accuracy that can be expected from this calibration is ± 1 ppb. The stability of this technology over time will be discussed below.

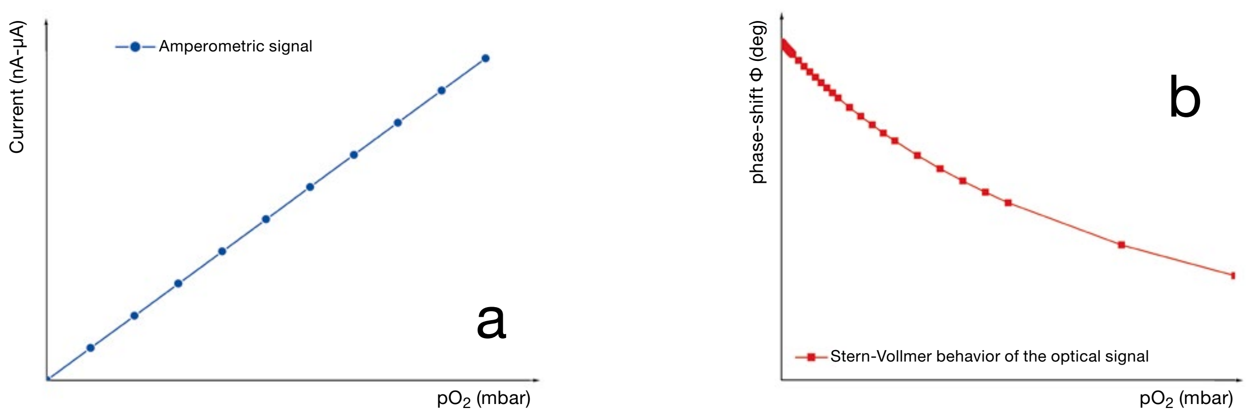

Figure 1: Differences of raw signal behavior against the content of oxygen with both sensors

Calibration

Whereas the Orbisphere amperometric sensor only requires a simple one-point calibration in air to determine the slope due to its "true zero," most amperometric sensors require regular zero point and slope calibration. As previously mentioned, the parameter that varies the most with optical technology is the value in the absence of oxygen. Since the other parameters defining the phase-shift normally show negligible change over time, the key parameter to be adjusted is the zero.

The calibration requires a specific setup, a specific calibration sample and delivers an accuracy of ± 1 ppb. Together with the factory defined parameters describing the curve at high oxygen levels, the overall accuracy is in general around ± 1 ppb or ± 2% of the measured value (whichever the greater).

Stability Over Time

All measurement devices drift with time. Hence, calibration is required at given intervals; the lower the drift the longer the interval between service and/or calibration. With the exception of the Orbisphere amperometric design that has no drift of the zero, all other amperometric sensors experience drift of both the zero point and of the slope – regular recalibration is therefore a necessity. In beer applications, this recalibration frequency is typically in the 1-3 months range for other amperometric sensors, whereas the Orbisphere sensor only requires a calibration in air during the bi-annual maintenance.

Existing optical systems claim to require calibration once every two years.[3] The conditions to achieve this two year frequency are that the system runs for only 12 hours per day; therefore it is switched off for the remainder of the time and is configured to provide data points every 30 seconds. The reality, however, is for systems to be configured to operate continuously and provide data points every 5 seconds. This results in a slight drift over time that will need to be addressed with a new calibration or an offset adjustment every six months.

Response Time

The response time of an amperometric sensor is determined by the permeability of oxygen through the measurement membrane. For sensors used in beer processes, 90% of the sample change is typically detected in 30 to 60 seconds. Furthermore, sensors using a guard electrode that prevents the effect of the oxygen present in the sensor electrolyte show an improved response time (up to twice as fast) towards low oxygen values. A response time (t90) from air to zero of 10s has been reported for optical sensors in a recent paper.[3] This is only true in the gas phase where the N_2 gas pushes the oxygen out of the luminescent spot (dye matrix).

Measurement data published recently by the Weihenstephan Research Center for Brewing indicates a faster response to a change towards higher oxygen containing beer for the Orbisphere amperometric sensor (t90 = 45 s) than for another optical system that was used (t90 = 70s).[4]

Maintenance Requirements

Whereas the maintenance of the amperometric sensor has long had the reputation of being tedious, most modern sensors are fairly easy to clean and refurbish. The Orbisphere A1100 sensor is supplied with patented pre-mounted membrane kits that include pre-dosed electrolyte that reduces bi-annual maintenance to only 3 minutes. The maintenance of an amperometric sensor has to be performed at regular intervals because the sensor becomes dirty and the electrolyte is consumed.

Optical sensors do not require such maintenance and the sensor head is normally cleaned during the CIP process. The only real maintenance is the replacement of the optical spot every 1-2 years depending on the process conditions. Installed Orbisphere M1100 optical sensors have performed extremely well, offering a calibration interval of over 6 months when in continuous use and providing a data point every 5 seconds (without the need to switch off the instrument when no beer is flowing through the pipe). In addition, the measurement range of the M1100 has been extended and now covers both high and low wort.

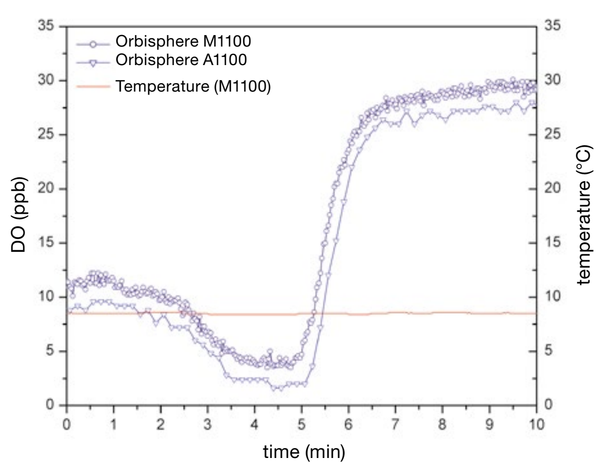

Figure 2: Sensor measurement response time

Conclusion

In beer, the oxygen exchange between the sample and the luminescent spot as well as accurate temperature measurement are keys to provide a fast response time.

We have proven that the latest optical sensor for beer has a response time equivalent to that of amperometric sensors in the brewing process (see Figure 2). In addition, the oxygen levels measured correspond well to values from the amperometric sensor (within less than 3 ppb).

Orbisphere M1100 Optical Dissolved Oxygen Sensor

Orbisphere A1100 Amperometric Dissolved Oxygen Sensor

References

1. Dunand F.A., Ledermann N., Hediger S., PowerPlant Chemistry 2006, 8(10), p.603

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3. Verkoelen F.; Brewing and Beverage Industry International, 2007, N° 1, 16.

4. Titze J., Walter H., Jacob F., Friess A., Parlar H.; Brewing Science, 2008, March/April, 66.

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HACH COMPANY World Headquarters: Loveland, Colorado USA

United States:	800-227-4224 tel	970-669-2932 fax	orders@hach.com
Outside United States:	970-669-3050 tel	970-461-3939 fax	int@hach.com

hach.com

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