Application Solutions

METTLER TOLEDO
Process Analytics

Oxygen Measurement Theory

Quality Measurements for
In-line Applications

Operating Principles for
Optimal Performance

Good Operating Procedures
for Optical Dissolved Oxygen Sensors

METTLER TOLEDO

Contents

1 Background

Oxygen, making up one-fifth of the Earth's atmosphere, is one of the most important elements. The combination of oxygen with other substances is the means by which energy is released in living cells. However, life can only be supported when the concentration of oxygen is within certain limits, hence the importance of oxygen measurements. Oxygen was not identified in science until 200 years ago, while methods of analysis have only been available since the last century.

Today, there are many methods and technologies employed for the measurement of oxygen in both aqueous and gas-phased media utilizing a membrane-based technology. METTLER TOLEDO offers two primary technologies, amperometric and optical, for the detection of oxygen. Amperometric technology has successfully been applied for decades in a variety of industries and applications. It has stemmed directly from the efforts of Dr. Clark and early polarography to develop into a robust and reliable technology. Amperometric technology is prevalent throughout the Biotech industry and is the primary technology employed for measurements in fermentation. Optical technology for oxygen detection is relatively new but has gained wide acceptance in the Biotech and Beverage industries. Both technologies are reliable and are used for applications across all types of process industries. Optical, however, is gaining more acceptance and use in Biotech applications because the technology requires less maintenance resulting in longer term reliability than amperometric systems.

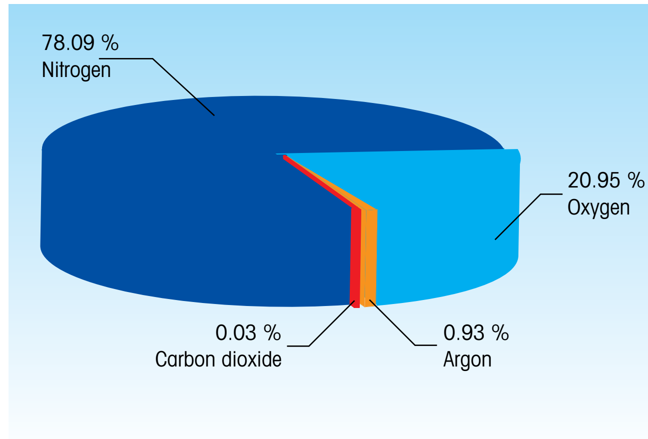


Figure 1: Atmospheric gas concentrations

This document focuses on Good Operating Procedures for METTLER TOLEDO's InPro 6860i Optical Dissolved Oxygen sensors optimized for Biotech fermentation processes. It includes an overview of the measurement technology, factors impacting measurements, an introduction to the benefits of METTLER TOLEDO's Intelligent Sensor Management (ISM®) technology, and common troubleshooting procedures. It is intended to assist owners and operators with developing best practices for operating and servicing their InPro 6860i systems but can also be applied as a general guide to all optical dissolved oxygen measurements in Biotech fermentation applications.

2 Oxygen Measurement Theory

2.1 Oxygen Partial Pressure

According to Henry's law, the amount of a given gas dissolved in a given type and volume of liquid is directly proportional to the partial pressure (p) of that gas in equilibrium with that liquid (reference Figure 2). This is an extremely important factor for all oxygen measurements. Understanding partial pressure's impact is critical to successfully monitoring oxygen in both liquids and gases.

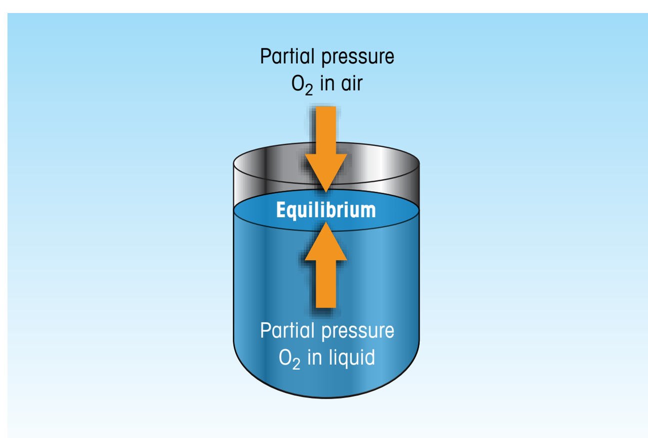


Figure 2: Example of Henry's law

For example, air is a mixture of gases in which oxygen contributes about 21% of the total pressure. Consider a volume of air at atmospheric pressure, 760 mmHg. 21% of this pressure (160 mmHg) is contributed by oxygen. This is the partial pressure of oxygen (pO_2) in this mixture.

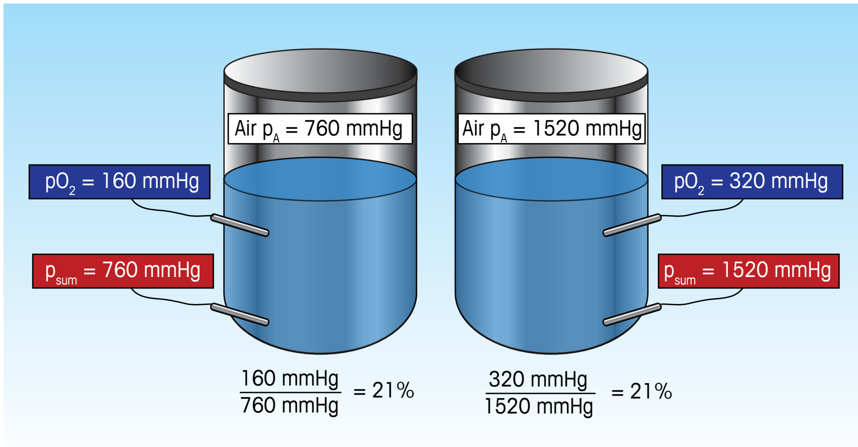


Figure 3: Relationship of pO_2 and O_2

As shown in Figure 3, if total pressure on this system is doubled to 1520 mmHg, the partial pressure also doubles to 320 mmHg. The relative percent of oxygen remains 21%. However, the sensor and transmitter would see a twofold increase in the oxygen concentration since the sensor is only responding to the partial pressure of oxygen. Therefore, it is extremely important for the oxygen transmitter or system to correct for system pressure in order that accurate measurements are made.

Conversion of oxygen partial pressure values to percent oxygen at other pressures is possible as follows:

$$\% O_2 = \frac{\text{Measured } \%O_2 \times pA}{\text{System Pressure}}$$

Based on Henry's law, partial pressure influences oxygen sensors in systems that contain compressed gases in headspaces of monitored vessels. Closed systems completely filled with liquid do not require compensation for system pressures.

2.2 Saturation vs. Concentration

Saturation (%Air, %O_2) is relative oxygen measurement expressed in percent comparing the reading to the expected equilibrium measurement within the gas phase. As per Henry's law, pO_2 increases as system pressures increase. However, the actual percent saturation value does not increase. The measurement requires pressure compensation in order to obtain accurate percent saturation readings in systems with dynamic changes to head pressures. Without pressure compensation, measured values would change with changes in system head pressures even though the absolute percent O_2 may not have changed.

Concentration (ppm, ppb, mg/l) gives the absolute amount of oxygen in a liquid during measurement. The concentration level of oxygen changes with changes to head pressures. As such, no pressure correction is needed.

Liquid column pressures in and of themselves have little practical impact on oxygen measurements up to 100m. However, in fermentation processes typical aeration is performed from the bottom of the vessel. In these cases oxygen partial pressure in the liquid is correlated to the pressure inside the air bubble which in turn is impacted by column pressures. Since saturation is a relative measurement, the impact of air bubbles on liquid column pressure is best compensated in optical dissolved oxygen measurements through a process scaling routine as found in Section 4.2.4 of this document.

Note: METTLER TOLEDO transmitters allow for manual entry of system pressure for compensation. Additionally, some transmitters may be configured with an external pressure sensor's analog signal (4-20 mA) or digital signal over a bus network for dynamic compensation of headspace gases and system pressures.

2.3 Solubility – Oxygen Concentration

Gases are soluble in liquid to varying degrees. This solubility, expressed as a mole fraction, is proportional to the partial pressure of the gas over the liquid (Henry's law). For most dissolved oxygen applications, the desired units of oxygen concentration are parts per million; ppm (when liquid density is 1 g/cm^3, ppm equals mg/l O_2). The mole fraction is easily converted to these units. However, oxygen measurements depend upon the oxygen partial pressure and the oxygen permeability of the membrane, but not on the oxygen solubility in the solutions. The oxygen concentration in mg O_2/l (CL) cannot therefore be determined directly with a sensor. As mentioned above, according to Henry's law the oxygen concentration is proportional to its partial pressure (pO_2).

CL = pO_2 X a
a = solubility factor

If "a" is constant, the oxygen concentration can be determined by means of the sensor. This applies at constant temperature and with dilute aqueous solutions such as drinking water. The solubility factor is strongly influenced not only by the temperature but also by the composition of the solution:

Medium, sat. with air	Solubility at 20 °C (68 °F) and 760 mmHg
Water	9.2 mg O_2/l
mol/l KCl	2 mg O_2/l
50 % Methanol-water	21.9 mg O_2/l

Although the solubility may vary widely, a dissolved oxygen sensor will give the same reading in all three solutions. Thus, determination of the oxygen concentration is only possible with constant and known solubility factors "a". Solubility may be determined by a Winkler titration or the method developed by Käppeli and Fiechter.

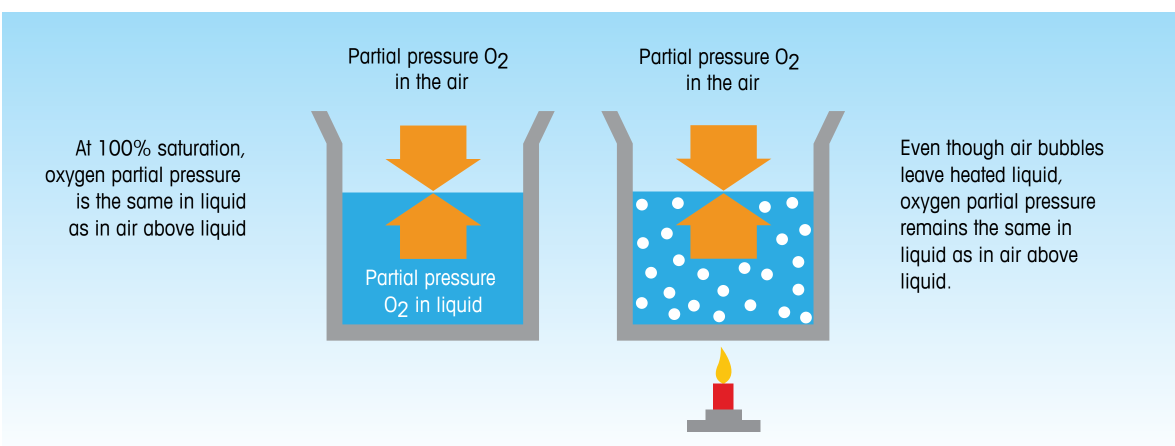


Figure 4: Relationship between %sat and concentration

2.5 Optical Oxygen Measurement

Optical oxygen sensor technology is based on an optical detection method often referred to as "fluorescence quenching". In contrast to the amperometric Clark-electrode, which detects a redox reaction of oxygen at the electrode, the optical method is based on an energy transfer between a fluorescing chromophore (fluorophore) and oxygen.

A fluorophore, embedded in the sensor tip, is illuminated with green-blue light from an LED source. The fluorophore absorbs the energy and transfers some of that energy in the form of an emitted red-fluorescence light. A detector in the sensor reads the emitted fluorescence and measures for intensity and lifetime. (Reference Figure 5 for an example of an optical sensor cross-section)

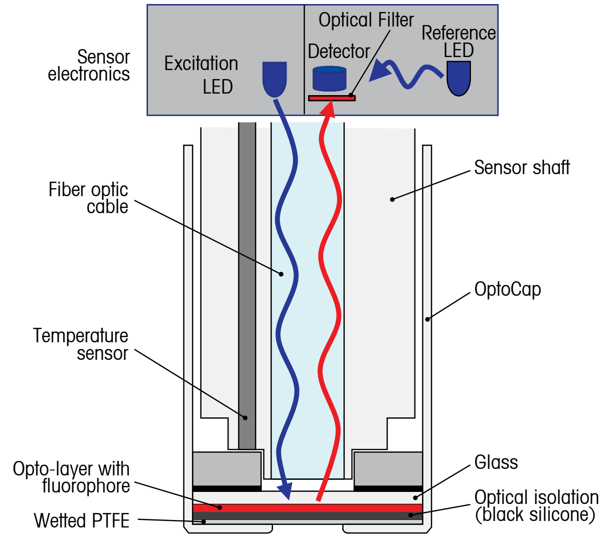


Figure 5: Optical sensor cross-section example

- In the presence of oxygen, the fluorophore transfers some of the absorbed energy to the oxygen molecules. Oxygen absorbs the energy and transfers it as heat to the surrounding area causing a change in fluorescence intensity and lifetime. The total fluorescence intensity and the lifetime are directly related to the oxygen partial pressure in the medium and are the basis from which accurate measurements are made.

- To analyze the fluorescence lifetime, a reference LED is used to compare measurements with those taken from the fluorophore layer. The excitation reference LED is pulsed with a constant frequency, the emitted fluorescent light shows the same pulsing but with a time delay to the excitation. This delay is called Phase shift or Phase angle and is referred to as "Phi".

- Phi is dependent on the oxygen level and follows a Stern-Vollmer correlation. Sensors are thus able to calculate oxygen concentration from measured Phi. Higher oxygen concentrations absorb more energy resulting in less energy to excite the fluorophore. This in turn causes a smaller phase shift than samples with lower oxygen concentrations. Figure 6 compares Phi measurement values at high and low oxygen concentrations.

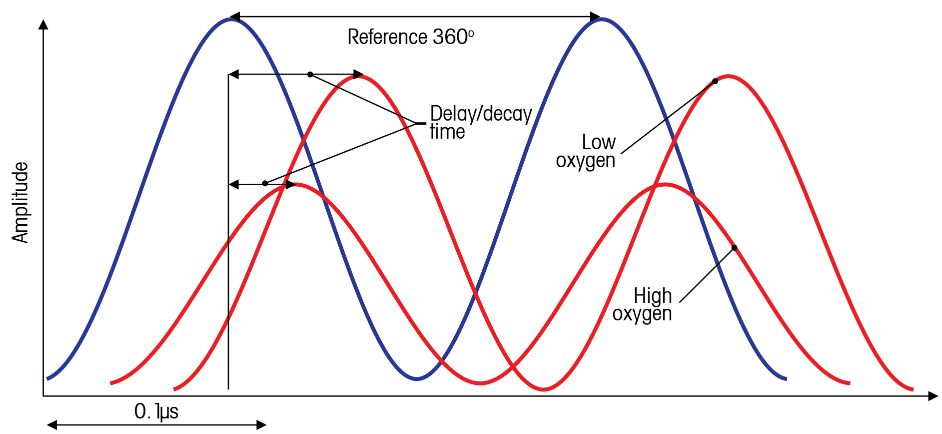


Figure 6: O_2 Phase shift measurement

- As previously mentioned, optical oxygen measurements monitor fluorescence decay time (Phase) of the fluorophore layer when flashed with an LED. The Phase is compared to a referenced measurement and calculated as a phase shift known as Phi. The changes in Phi between various concentrations are not linear, and are calculated using a Stern-Volmer equation once an initial two points are known. The results, when plotted, produce a calibration curve.

2.6 Temperature

Temperature impacts the rate of degradation of the fluorophore with higher temperatures accelerating the rate of decay. The rate of degradation is further accelerated when optical sensors are making active measurements in elevated temperatures. As such, oxygen measurements should not be made through CIP and SIP conditions.

2.7 Dependence on Flow

Unlike amperometric sensors, optical oxygen sensors are not flow dependent because oxygen is not consumed during the measurement.

2.8 Sensor Design

InPro 6860i sensors consist of a sensor head containing the electronics and LED source, a sensor shaft with fiber optic cable, and an OptoCap containing the optical fluorophore layer and a gas-permeable membrane. Sensors are powered from a transmitter's or external 24 Vdc power source.

The oxygen molecules migrate through the membrane in the OptoCap and absorb light energy from the LED light source. The energy absorbed from the presence of oxygen reduces the amount of energy available to fluorescently excite the fluorophore. The reduction of excitement is measured and used to calculate the amount of oxygen present in the media.

InPro 6860i optical sensors utilize a wetted PTFE membrane over a relatively thick silicone membrane. Both are highly permeable to oxygen molecules. The fluorophore layer embedded in an organic matrix sits behind the silicone. The PTFE and silicone layers protect the fluorophore layer from being directly wetted by the media and also from the diffusion of other chemicals from the sample solution into the fluorophore layer.

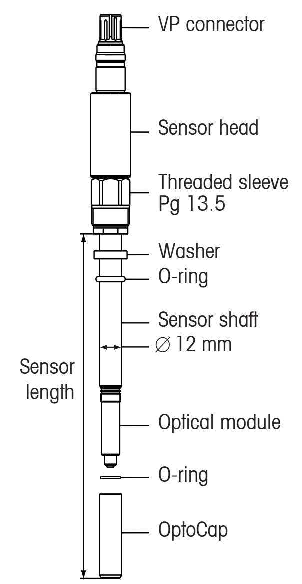


Figure 7: InPro 6860i sensor design

3 Communication Installation Options

InPro 6860i sensors provide both RS485 digital communications and simulated amperometric oxygen sensor operation. RS485 communication is possible with select transmitters from METTLER TOLEDO and is ideal for new installations. Simulated amperometric operation, on the other hand, enables InPro 6860i sensors to be retrofitted into existing installations or connected to a wide variety of biocontrollers.

3.1 Digital RS485 Communication

RS485 communication provides optimal performance and maximum functionality by digitally communicating with select METTLER TOLEDO transmitters. The 2-way communication between sensor and transmitter takes full advantage of the advanced performance inherent with systems endowed with Intelligent Sensor Management (ISM). ISM provides, among other features, "Plug & Measure" operation with oxygen measurement available immediately after installation. Sensors are recognized automatically and all relevant data are uploaded to the transmitter. Please reference Section 7.0 of this document for a more complete discussion on the capabilities of ISM for the InPro 6860i.

Additionally, select METTLER TOLEDO transmitters, with the exception of those powered from digital bus networks, provide power to the sensors, greatly simplifying the installation. Bus-powered METTLER TOLEDO transmitters require InPro 6860i sensors to be powered with 24 Vdc, 800 mW independent of the bus.

3.2 Simulated Amperometric Communication

InPro 6860i sensors simultaneously generate a nA current for O_2 and NTC 22 KOhm signal for temperature measurements. Simulated signals are used to retrofit sensors into existing installations or provide functionality with the majority of biocontrollers found throughout Process Development (PD) facilities. The vast majority of biocontroller or retrofit installations require InPro 6860i sensors to be powered with 24 Vdc, 800 mW independently. METTLER TOLEDO offers a variety of cable and adapter options to facilitate powering of sensors.

VP8 Cable			METTLER TOLEDO ISM Transmitter			Non-ISM Transmitter or Biocontroller
			M400	M800 CH1	M800 CH2	
Callout	Color	Function	TB4	TB2	TB4	Transmitter function
A	black/transp.	cathode (nA)	not used	not used	not used	cathode (nA)
B	red	anode (nA)	not used	not used	not used	anode (nA)
C	gray	sensor (+)	1	9	9	sensor (+)
D	blue	sensor (-)	2	10	10	sensor (-)
E	white	NTC 22kΩ	not used	not used	not used	NTC 22kΩ
F	green	NTC (GND)	not used	not used	not used	NTC (GND)
G	pink	RS485 (+)	8	14	14	not used
H	brown	RS485 (-)	7	13	13	not used
S	green/yellow	shield	4	12	12	shield

InPro 6860i VP Connection

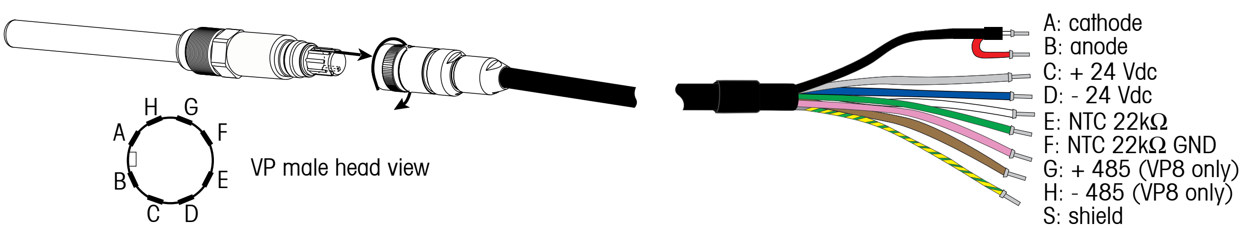


Figure 8: InPro 6860i installation wiring options

4 Sensor Calibration

As mentioned in Section 2, the calculation of the Phi value at various oxygen concentrations, most often 100% air (Phi 100) and 0% (Phi 0), is the basis of sensor calibration. Optical sensor calibrations establish Phi values for known concentrations and calculate remaining points with a Stern-Vollmer equation. Results when plotted are a calibration curve similar to what is shown in Figure 9, from which accurate measurements are possible.

4.1 Calibration Requirements

Each OptoCap has unique Phi values for Phi 100 and Phi 0 necessitating an initial 2-point calibration. New

InPro 6860i sensors are 2-point calibrated at the factory and do not need an additional 2-point calibration prior to operation.

Important considerations for calibration:

- For calibration in gas (air) the OptoCap must be dry, since adhering water drops can falsify the measured oxygen value.
- Make sure temperature and pressure values are constant.
- Calibration always needs accurate pressure and temperature measurement. Only process scaling is independent of pressure (see Section 4.2.4).
- Make sure that the correct calibration pressure, humidity and salinity values are set in the transmitter before the calibration is started.
- In the event of calibration in water or sample medium, the calibration medium must be in equilibrium with the air. Oxygen exchange between water and air is very slow. Therefore, it takes quite a long time until water is saturated with atmospheric oxygen.
- Calibration in a fermenter post-sterilization should only be performed as process scaling unless process pressures are accurately accounted for (see Sections 4.2.3 and 4.2.4).
- Please refer also to the transmitter manual for detailed information.

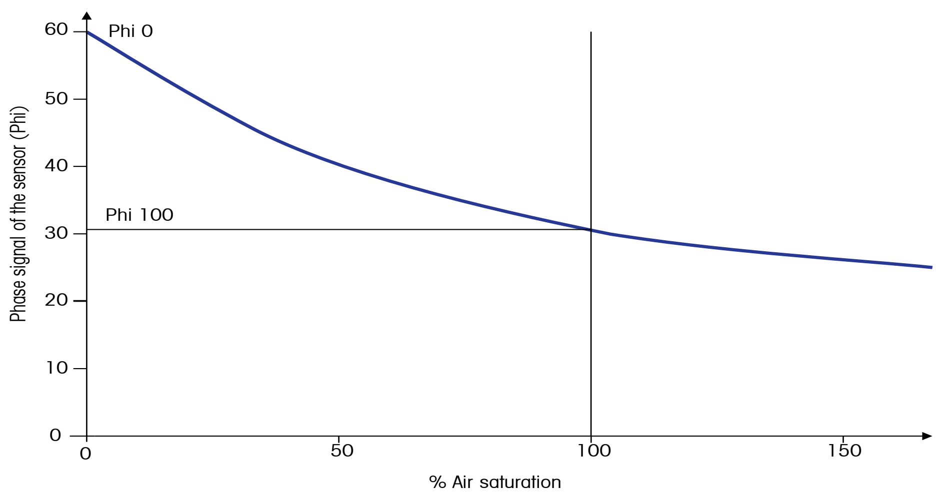


Figure 9: O_2 calibration curve

4.2 Calibration Types

4.2.1 2-Point Calibration

A 2-point calibration establishes the Phi 100 and Phi 0 unique to the OptoCap. As previously discussed, a 2-point calibration is required with every OptoCap exchange. For correct calibration, use nitrogen gas or other oxygen-free medium with a purity level of at least 99.99% to achieve the Phi 0 point, followed by exposure to air for the Phi 100 point. 2-point calibrations are performed on METTLER TOLEDO transmitters or on computers with iSense™ software. The resulting calibration curve is stored on the sensor upon completion and is continuously referenced as the sensor makes its measurements.

4.2.2 1-Point Calibration

1-point calibrations establish a new Phi 100 value. Sensors reference the new Phi 100 value against the stored calibration curve to create a new calibration curve. 1-point calibrations can calibrate sensors in air with measurement settings at 100% air, 20.95% O_2, or 8.26 ppm and local atmospheric pressure (see instruction manual for the transmitter). 1-point calibrations are performed on METTLER TOLEDO transmitters or on a computer with iSense™ software.

4.2.3 Process Calibration

Similar to 1-point calibrations, process calibrations establish a new Phi (Phi 100 or Phi 0, depending on the oxygen level during the calibration) value from which a new calibration curve is calculated. Process calibrations differ in that they are generally conducted with the sensor in-situ in a vessel or piping system. Because calibration curves are not linear, 1-point process calibrations in vessels with headspace must accurately account for system pressures or risk jeopardizing the accuracy of the entire curve. Because of this, METTLER TOLEDO recommends using 1-point process scaling instead of process calibration for the majority of post-SIP applications.

4.2.4 Process Scaling

Unlike 1-point calibrations or process calibrations, process scaling (i.e. scales) sets the the measurement value to a desired level without making any adjustments to the calibration curve. This procedure is recommended for most biotech applications after post sterilization (autoclaving) or SIP with systems set to an initial value.
The phase values of the sensor are not adjusted, only the displayed values and the nA outputs are rescaled to the desired value. In this manner, the processed scaled 100% value most closely resembles the historic 100% value of amperometric sensors. Reference Figure 10 for a process scaled curve in a vessel with some head pressure. Note the original calibration curve is not adjusted, just the scaling of the output is set to 100%. With this method the real process pressure and the solubility factor for oxygen can be ignored.

4.2.5 Simulated Amperometric Standardization

As discussed in Section 3.2, InPro 6860i sensors for biocontroller or retrofit installations use a simulated nA current for oxygen measurements. The nA current is linearly proportional to the calibrated Phi 100 and Phi 0 with ~65 nA set for 100% at 25 ºC and 0 nA set for 0%. Standardization of biocontrollers and non-ISM-enabled transmitters simply follow the calibration/standardization recommendations for amperometric sensors.

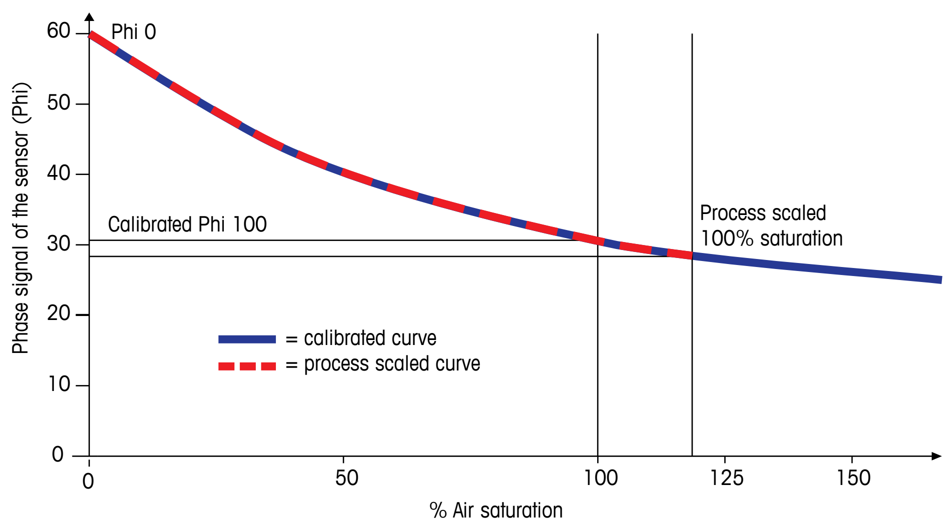


Figure 10: Process scaled curve example

5 Stability Control and Calibration Recommendations

Actual Phi 100 and Phi 0 shift with time and operation is shown in Figure 11. The shift is caused by normal and expected degradation of the fluorophore. Phi values change as part of the degradation process with the amount of change greater at Phi 0 than at Phi 100.

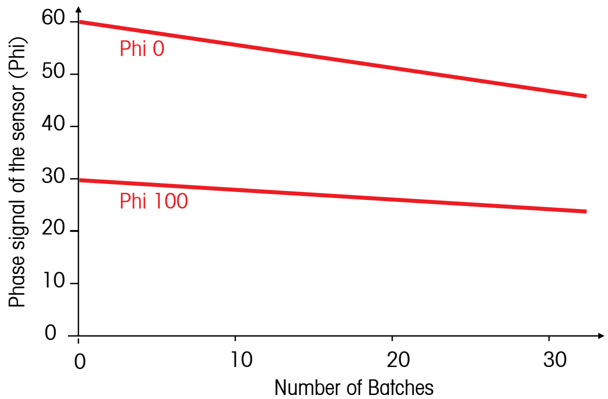


Figure 11: Normal Phi 100 and Phi 0 shift

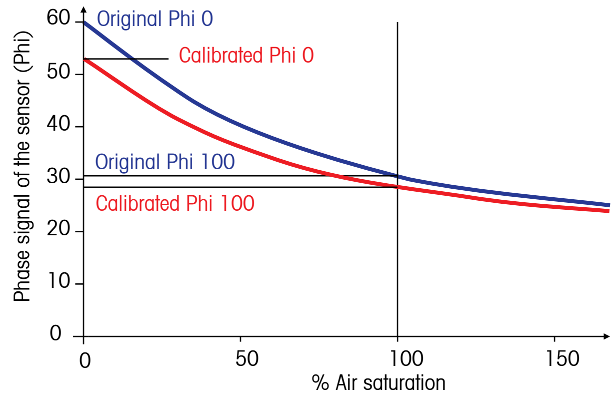


Figure 12: Normal calibration curve shift

The rate of degradation is primarily influenced by the measurement itself. During the measurement the LED sample rate, oxygen value, and process temperature directly contribute to the rate of degradation. High sample rates in high oxygen media with elevated temperatures quickly degrade the optical dye, causing the greatest shift in the Phi values. It is important to note, an active LED is the primary contributor of degradation. High oxygen levels and high temperatures do not significantly contribute to degradation without the LED being active. Thus, as long as the sensor is switched off, e.g. during sterilization, the degradation is extremely small. Figure 12 is representative of an expected calibration shift due to normal degradation of the fluorophore. To prolong the lifetime of an OptoCap and to increase the long term stability, please use the longest possible measurement interval and the different LED switch off functionalities (see Section 7.5 for LED control details).

5.1 Stability Control

The degradation of the fluorophore is very linear over the lifetime of an OptoCap. The purpose of periodic calibrations is to correct for the normal sensor aging. As shown in Figure 13, calibrations adjust the values to provide accurate readings. METTLER TOLEDO is able to offer dynamic compensation of sensor aging to limit its impact on the accuracy of measurement.

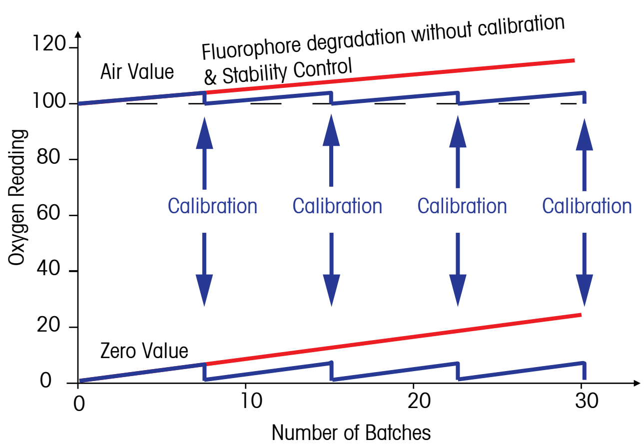


Figure 13: Calibration adjustments without Stability Control

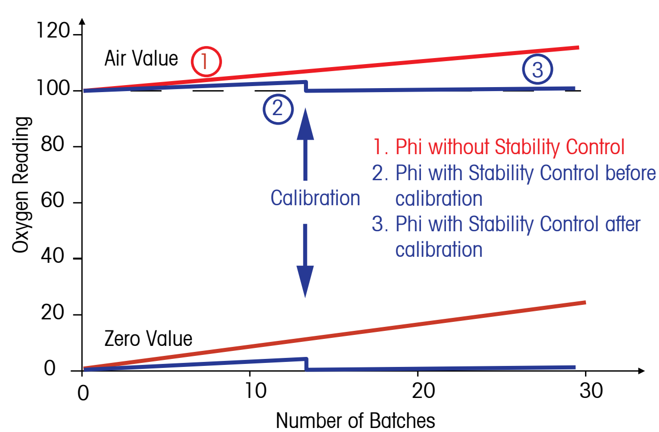


Figure 14: Calibration adjustments with Stability Control

InPro 6860i sensors utilize a proprietary algorithm that monitors the sample rate, oxygen level and process temperature to accurately calculate shifts in Phi values caused by degradation. METTLER TOLEDO terms this compensation, Stability Control. As implied by the name, Stability Control stabilizes the oxygen reading and significantly reduces the need for frequent calibrations.

This stability control is able to learn process specific sensor aging. By performing a 1-point calibration in air after some batches, the sensor compares calculated Phi shift values with calibration Phi shifts to most accurately compensate for future fluorophore degradation. As a result, there is nearly no drift of the sensor in the process, as shown in Figure 14.

5.2 Recommended Calibration Frequency

InPro 6860i systems must have an initial 2-point calibration upon exchange of OptoCaps. This is a common requirement for all optical-based oxygen technologies. The 2-point calibration process establishes the initial Phi 100 and Phi 0. Subsequently, only periodic 1-point calibrations with infrequent 2-point calibrations are required on systems with Stability Control. The Stability Control algorithm provides accurate compensation for aging.

In general:
A 2-point calibration is required:
- For each new OptoCap
- If high accuracy is needed below an oxygen partial pressure of 60 mbar

A 1-point calibration is sufficient:
- As long as the oxygen level is above 60 mbar partial pressure

Frequency of calibration is best determined by duration of the process batches and frequency of sterilization cycles. Normal 3 to 5-day batch processes should wait at least 5 batches for the initial 1-point calibration to provide the Stability Control algorithm with sufficient process condition data. Additional 1-point calibrations are not likely required for another 5 to 10 batches. 1-point calibrations or 2-point calibrations in applications measuring below 60 mbar prior to commencement are recommended in processes of longer duration such as fed-batch and perfusion methods.

Some facilities may find it difficult to accurately track the number of batches their InPro 6860i sensor inventory has been exposed to. An alternate and perhaps more practical method of determining when to conduct 1-point calibrations may therefore be strictly time based. Systems with METTLER TOLEDO transmitters have the option to utilize ISM's Adaptive Calibration Timer (ACT) parameter (see Section 7.2) to indicate recommendations for when to calibrate. The ACT can be configured around the average duration for 5 to 10 batches to occur in a manufacturing facility. The ACT can be dynamically monitored over a 4-20 mA signal, a relay, or an Analog Input (AI) of a FOUNDATION fieldbus™ or PROFIBUS PA transmitter. Another approach would be simply date based. For example, a facility may choose to calibrate every sensor on the first week of every other month.

6 Challenges with Optical O_2 Measurements

As with any sensing technologies, accurate measurements may be impeded by a variety of factors. The cause of impedance often varies and may be a combination of issues. Generally, the issues can be divided into three categories: Sensor Related, Media Related, and External Related.

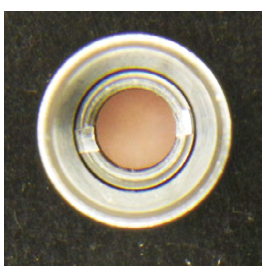
6.1 Sensor Related Issues

Sensor related issues may be caused by improper calibration or sensor setting. Improper calibration and bad zero offset will impact the ability of a sensor to provide reliable measurements.

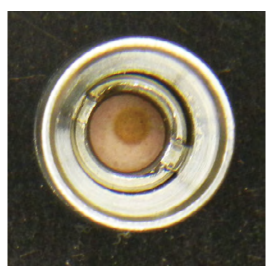
Errors resulting from process calibrations conducted in vessels with headspace pressures are common occurrences. It is very important to understand and review the section on Partial Pressure in this document. Many operational challenges to accurate and reliable DO measurements stem from not accounting for the headspace pressures during process calibrations. METTLER TOLEDO recommends the use of process scaling in place of process calibrations for the majority of applications. The differences between these two methods are outlined in Sections 4.2.3 – 4.2.4. If process calibration is required, ensure system pressures are correctly input for on-line measurements to most accurately match any grab samples.

6.1.1 OptoCap Wear and Damage

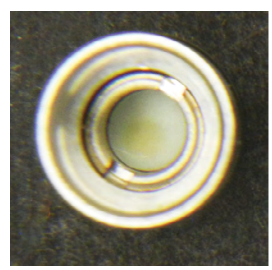
Normal OptoCap wear is the most common sensor related measurement issue. Depending upon operating process conditions and LED sample rates, OptoCaps can have an operational lifetime of up to 360 days. However, OptoCaps do wear and require periodic replacement. Figure 15 provides examples of how new, worn and bleached OptoCap fluorophores appear. The sensor continuously monitors if OptoCap performance (Phi values and intensity) is within acceptable levels. Systems found to be out of tolerance return error messages during transmitter operation or system calibrations with iSense. OptoCaps should be replaced in the event this occurs.



New OptoCap fluorophore



Depleted OptoCap fluorophore from standard operation



OptoCap fluorophore bleached by chemical attack or high temp. operation

Figure 15: OptoCap fluorophore examples

Symptoms of worn OptoCaps or damaged sensors can be displayed during system response testing. For example, symptoms could include sensors not responding to zero within the specified time or not reaching zero at the low end of measurement. Please refer to the Response Testing section (8.1) of this guide for more information.

Physical damage to the sensor can also be a cause of sensor related issues. The InPro 6860i is a rugged sensor capable of withstanding SIP and autoclave cycles. However, it contains a fiber optic cable in the sensor shaft that, although extremely uncommon, can crack if the sensor is improperly handled or dropped. The primary symptom of a cracked fiber optic is highly erratic and unstable readings resulting from an inconsistent transmission of light energy to and from the fluorescent fluorophore layer. Sensors with suspected cracked fiber optics cannot be repaired and will require replacement. Suspect sensors should be sent to METTLER TOLEDO for evaluation.

6.2 Media Related Issues

The fluorophore dye of the InPro 6860i has been optimized for biotech fermentation processes. Chemical substances not common to biotech processing may chemically react with the fluorophore accelerating its degradation (H_2S, Cl_2, other strong oxidizing chemicals). This is especially true if the sensor is sampling during exposure to chemicals. OptoCaps are compatible with most Clean in Place chemicals as long as the LED is shut off during these operations.

Media attack on OptoCaps will generally result in the depletion or "bleaching" of the fluorophore. Sensor signals will spike past expected values and rise above 100% scale or completely over range if the dye is sufficiently bleached. Removal of the contamination from the media, ensuring the LED is turned off during periods of chemical exposure, and/or replacement of the OptoCap will likely correct the issue.

6.3 External Related Issues

6.3.1 Trapped Air

Gas bubbles with higher pO_2 than the liquid may cause an increase in rates of O_2 penetration through the membrane, resulting in a measurement that is greater than actually present in the media. This occurrence is most common in sensors vertically installed in the head plate of small reactors with sparger systems. Care should be taken to ensure the sparger is not so near the sensor installation so as to affect proper functionality. For best operation, sensors should be mounted so that sensor tips are not subjected to direct exposure to sparger gas bubbles.

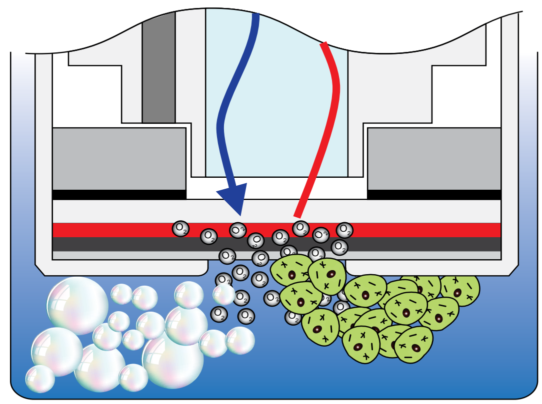


Figure 16: Impact of air bubbles and biological growth

6.3.2 Biological Growth

Biological growth on sensor membranes may occur in some installation. The biological growth will combine to coat the membrane and consume O_2 molecules, preventing them from entering the OptoCap. Symptoms of biological growth are displayed as a gradual decline in anticipated readings. Sensors should be mounted at an angle or even upside down in applications in which biological growth is expected to coat membranes. In extreme cases, operators may consider mounting sensors in the headspace and rely on Henry's law for the calculation of dissolved oxygen content.

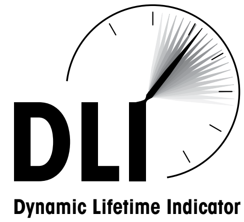
6.3.3 Varying System Pressure

InPro 6860i sensors measure the partial pressure of oxygen (pO_2). Vessels with fluctuating headspace pressures will result in fluctuating pO_2 measurements. METTLER TOLEDO transmitters have the ability to dynamically compensate for fluctuating system pressures.

7 Intelligent Sensor Management (ISM)

InPro 6860i sensors feature METTLER TOLEDO's ISM technology. ISM is a digital platform for in-line process analytical systems that simplifies sensor handling, enhances measurement integrity, and reduces sensor life-cycle costs. ISM sensors provide analog to digital measurement signal conversion, Plug and Measure start up, on-board data storage, and diagnostics algorithms.

7.1 Dynamic Lifetime Indicator (DLI)



The Dynamic Lifetime Indicator (DLI) is a diagnostic parameter providing information about the remaining reliable lifetime of the OptoCap. The DLI is expressed in units of Days with the predicted days of reliable operation remaining, counting down. As long as the DLI is above zero days the system is within the specified accuracy after a calibration. If the DLI is zero after a calibration, the OptoCap should be replaced. The DLI parameter may be monitored dynamically through a transmitter's analog output and relay or through an Analog Input (AI) of a bus-enabled transmitter. The DLI can also be associated with a transmitter alarm for indication when it falls below a set level.

Contributing factors for aging of the OptoCap and the counting down of the DLI include:
- number of measurements
- temperature during measurement
- oxygen concentration during measurement
- number of CIP cycles
- number of SIP cycles
- number of autoclavings

The DLI is calculated in two different ways:
1. Continuously: Using the above parameters actual sensor stress is calculated. With each measurement the sensor load is increased. The accumulated load divided by the elapsed time is the basis of the calculation of the remaining lifetime. Changes in process conditions like temperature and oxygen value are automatically taken into consideration.

2. During calibration: Calibrated Phi values are compared to the Phi value of the first calibration of the actual OptoCap. Using the above calculated sensor load and the elapsed measurement time since the first calibration, the remaining lifetime of the OptoCap is calculated. The calculation after a calibration gives a higher accuracy of the DLI compared to the continuous calculation. Thus the DLI value can be significantly different after a calibration. The longer an optical sensor is in use, the more accurate is the DLI information. Short term process changes with a new sensor influence the DLI much more than after a longer time in use.

Note: For a correct DLI calculation an accurate calibration is essential.

Note: For a correct DLI calculation the date and time must be set correctly in the transmitter before connecting a sensor.

Depending on the age of the OptoCap, the phase values typically decrease over time compared with a new OptoCap. The DLI continuously monitors the OptoCap Phi 100 and Phi 0 values to ensure they remain in specification as noted below:

	New OptoCap		Limit for old OptoCap	
InPro 6860i	Phi 0	Phi 100	Phi 0	Phi 100
	60° ± 2°	32° ± 3°	> 50°	> 15°

7.2 Adaptive Calibration Timer (ACT)

The ACT provides information as to when the next calibration is required to ensure measurements will remain within the specified accuracy. The initial calibration interval in days is user programmed with actual calculation based upon DLI information. Systems with high temperatures and LED sample rates will count down the remaining days to calibration more quickly than systems with lower temperatures and LED sample rates. The ACT parameter may be monitored dynamically through a transmitter's analog output or through an Analog Input (AI) of a bus-enabled transmitter. The ACT can also be associated with a transmitter alarm for indication when it falls below a set level.

7.3 Total Operation of OptoCap (tooCap)

Bus-enabled transmitters provide a continuous count in days of total time of operation of an OptoCap. The tooCap counts up in increments of days and gives operators a quick snapshot of how long a particular OptoCap has been in service, and provides useful trending information when compared with the DLI. The tooCap is an excellent parameter to monitor to ensure OptoCaps have been exchanged and calibrated before commencing with a product change or entering a long-term batch process. The tooCap parameter may be monitored dynamically through a transmitter's analog output or through an Analog Input (AI) of a bus-enabled transmitter. The tooCap can also be associated with a transmitter alarm for indication when it rises above a set level.

7.4 Calibration History

The last three calibrations and the factory calibration data are stored in the sensor's memory. This data can be read out with a transmitter or with iSense software. The calibration history gives valuable information regarding the quality of the calibration and the aging of the OptoCap.

7.5 LED Control

The primary contributing factor to the aging of an OptoCap is the measurement itself. To prolong the lifetime of the OptoCap, the measurement can be switched off if the system is not needed. Active measurement during CIP cycles or when the sensor is exposed to high oxygen levels during standby of the plant should be avoided. With the LED shut off, the sensor sends a constant measurement value of –1% air to the transmitter and the transmitter is set to the "Hold mode". To configure the "Hold mode" please refer to the transmitter manual.

7.5.1 Automatic Switch Off at High Temperature

If the LED mode is set to "Auto" (default setting) the sensor LED will be switched off as soon as a specific process temperature is reached. The default temperature for the InPro 6860i is 60°C/140°F but this limit can be set to an individual value below 60°C/140°F with a METTLER TOLEDO transmitter or with iSense software. Above 60°C/140°F the sensor will always be switched off. These settings are also active if the sensor is connected to the process as a simulated amperometric sensor. The switch off temperature should be set at least 5°C/9°F higher than the highest process temperature. For example, if the highest process temperature is 40°C/104°F, 45°C/113°F should be the minimum set-point. In this situation, as soon as the temperature exceeds 45°C/113°F the sensor will stop measuring and the LED will be switched off. For the sensor to switch on, a hysteresis of 5°C/9°F is implemented, meaning that the sensor (and LED) will be switched on as soon as the temperature drops to 40°C/104°F.

7.5.2 LED Switch Off of the Sensor

The sensor LED can be switched off manually through the transmitter menu by setting the LED mode to "off" or by a remote signal (digital input). To restart the measurement, the LED mode needs to be set manually to "on" via the transmitter menu, or via a remote signal (digital input). Select METTLER TOLEDO transmitters with FOUNDATION fieldbus™ and PROFIBUS PA also have the ability to switch the LED off and on through commands sent over the bus network.

7.5.3 Remote Switch Off through Hold Function

METTLER TOLEDO transmitters can be set to "Hold" by applying an external digital signal (see the transmitter manual). In this situation the sensor and the sensor LED are switched off. As soon as the "Hold Mode" is off, the optical sensor will continue to measure using the previous settings.

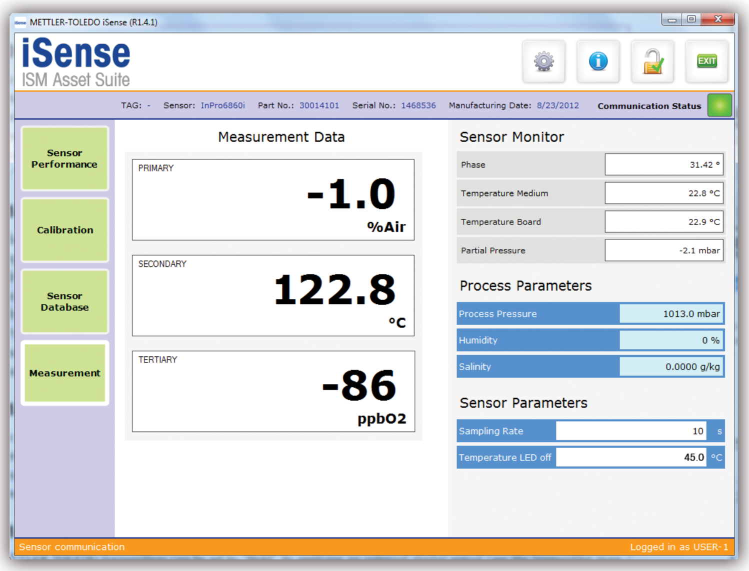


Figure 17: iSense software with InPro 6860i sensor connected and LED auto switch off set to 45 °C and active

8 Sensor Maintenance

InPro 6860i sensors are extremely reliable when properly calibrated and operated. Beyond periodic replacement of OptoCaps little to no maintenance is required. To replace the OptoCap you first have to unscrew the cap sleeve.

Attention! If the cap sleeve is detached, be aware of the fiber optic cable in the center of the sensor shaft. Damage and soiling of the optical fiber may influence the signal or destroy the sensor. Small soiling can be removed with a lint-free cloth.

To replace the OptoCap, the entire membrane cap is replaced.

- Unscrew the OptoCap from the sensor shaft and carefully pull it off the sensor.
- Place the new OptoCap over the sensor shaft and screw it into place.
- After each exchange of the OptoCap, reset the DLI manually using either a transmitter or iSense.
- Perform a 2-point calibration after each exchange of the OptoCap

Figure 18: OptoCap and InPro 6860i sensor shaft

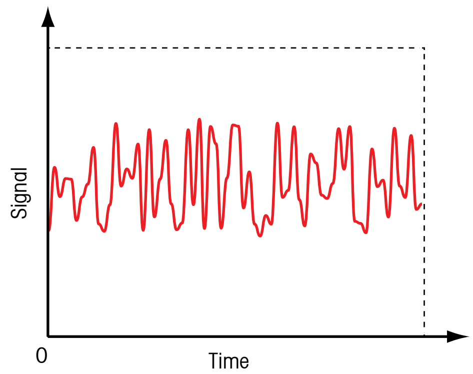
8.1 Response Testing

Response testing is the best means available to ascertain the proper functionality of InPro 6860i sensors. Response testing involves cycling an InPro 6860i sensor from an O_2-rich environment to an O_2-free environment.

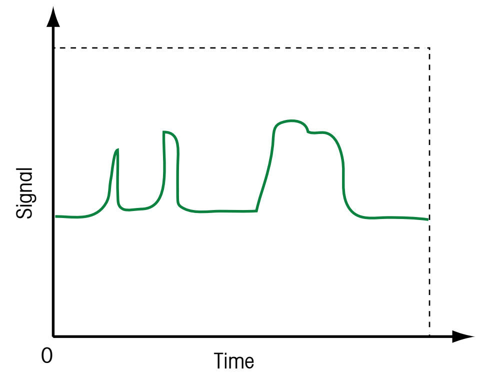
Good sensor specifications are:

Response time from air to zero in N_2: < 2% SAT after 70s
Response time from zero to air: > 98% SAT after 70s

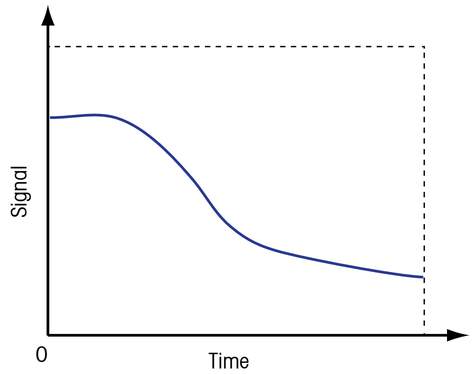
InPro 6860i O_2 Sensors
In Situ Troubleshooting Examples



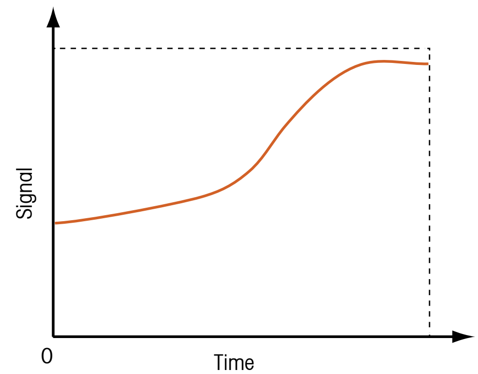
Erratic/Unstable Signal
Possible Causes: Mechanical Damage to Sensor or OptoCap Damage



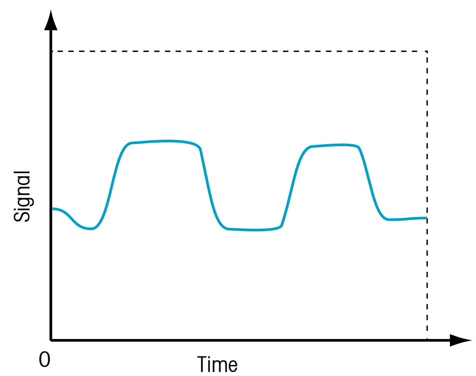
Spiking of Signal
Possible Cause: Air Bubbles Present in Media



Gradual Decline of Signal
Possible Causes: Membrane Coating or Bacteria Growth



Gradual Increase of Signal
Possible Causes: Increasing System Pressure without Compensation or Degraded OptoCap



Signal Spiking with Plateau
Pausible Cause: Fluctuating System Pressures Between Various Processes

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For more information

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Subject to technical changes

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