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Information on long-term stability and accuracy of Aanderaa oxygen optodes.

Information about multipoint calibration system and sensor option overview.

(2014-04-10, by Anders Tengberg and Jostein Hovdenes)

Summary: *This document is a summary of the latest findings concerning Aanderaa optodes and about the multipoint oxygen calibration system that was taken into operation in the autumn of 2012. In general Aanderaa optodes have proven excellent field stability; the longest continuous deployments have been more than 6 years without detectable drift. Frequently delivered sensors have however read low by several %. The main reason for this is that foils bleach both because of ambient light (especially fluorescent room light) and because of excitation light if sampling is done at high frequency. The bleaching process has more impact when foils are new. From the middle of 2012 methods are in place to pre-treat all foils that are used on multipoint calibrated sensors before these are mounted on the optodes and calibrated. With this treatment optodes experience an initial drift of less than 0.15 % lower readings per 100 000 samples. Foils become better with time and after 3.5 million samples this drift is typically around 0.025 % lower readings per 100 000 samples. New improved firmware (framework 3) and a red LED for internal referencing have been implemented in the more recent optodes (models 4835, 4330, 4831 and 4531). In the middle of 2013 we introduced the 100 m rated Aquaoptode (model 4531). It uses the same electronics and optics as the original sensors, but with new housing and form factor price has been significantly reduced, keeping the same reliability.*

Introduction: Any project that aims to study the in situ dynamics of oxygen, should select an accurate method with proven long-term field stability. Commercially available oxygen optodes for oceanographic application were introduced by Aanderaa Data Instruments A/S (today part of the Xylem group) in 2002. These sensors are, to the best of our knowledge, the only type of dissolved oxygen sensor with proven consistent long-term (years) field stability. Two factors making AADI optodes more stable and reliable than other oxygen sensors, electrochemical or optical, are:

1. Stable foil chemistry that withstands high temperature and high pressure
2. Rugged stand-alone sensors design with stable electronics and modern signal processing

In addition it should be noted that Aanderaa optodes are compact, consume little energy, have calibration constants and data-processing included in the sensor (smart-sensors) and do not need any additional devices (e.g. pumps) to work correctly.

Sensor long-term stability: Numerous published studies on sensor long-term stability are include in the publication summary below. The most complete study of accuracy and long-term performance of Aanderaa optodes was presented at the 2010 AGU meeting as a research poster by Takeshita et al. In this work recordings from 69 Aanderaa optodes deployed on Argo floats in different oceans were compared with data from the Open Ocean Atlas. The average drift of the sensors was found to be 0.01 ± 3.0 µM/year. The average accuracy was -16.72 ± 12.5 µM. An extension of this study was published in a scientific paper by Takeshita et al. (2013).

Fig 1: Optode 3830 (MkI)

In conclusion the optodes were stable but gave lower values than expected. The main reason for this is that foils bleach both because of ambient light, especially fluorescent room light, and because of excitation light if sampling is done at high frequency (see details below).

Bleaching assessment and recommended use of black protection cap at storage: In order to quantify and compensate for the light induced drift we have been running several optodes in an air saturated bath at 1 Hz sampling frequency in parallel with optodes sampling at 20 minutes interval. When analyzing these data we find that the optodes sampled at 20 minutes interval do not drift significantly over time periods of years. Optodes sampled at high frequency drift at different levels depending on if the foils were new and if they were pre-treated or not, see graph below (Fig. 2).

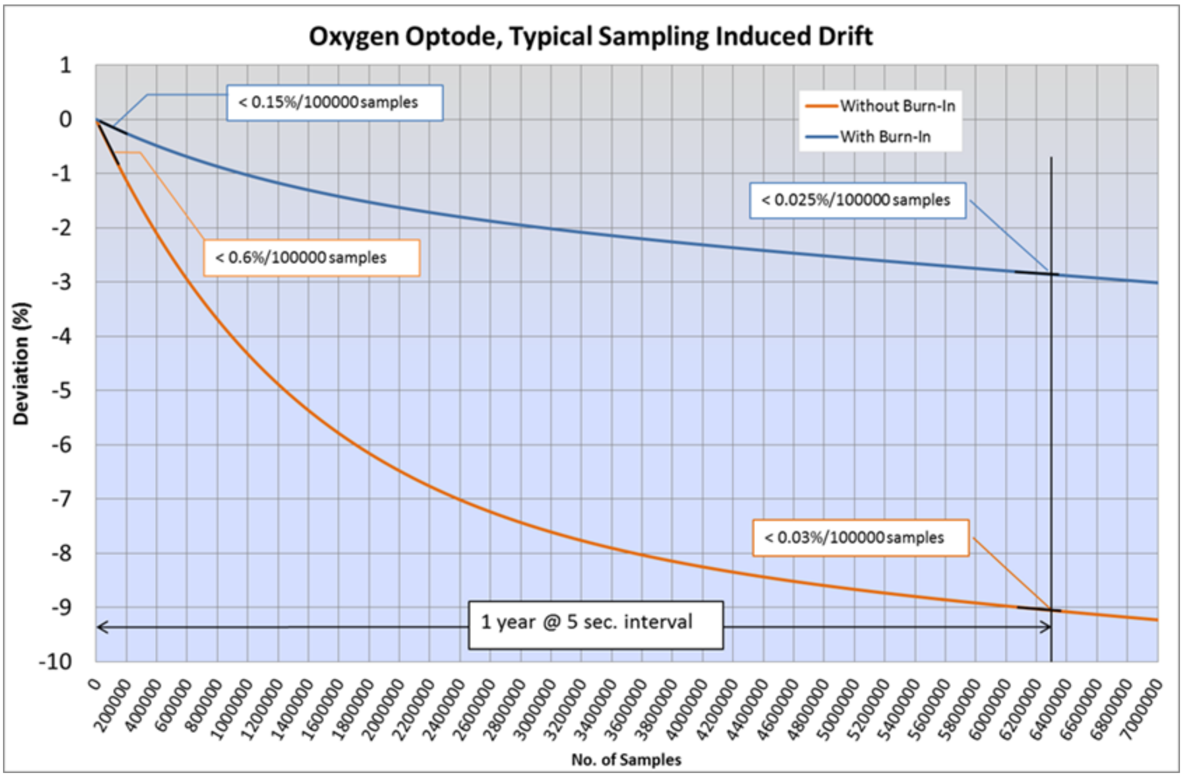

Fig. 2: Optode drift in relation to number of measurements and pre-treatment (with burn-in) or not.

We se that the initial slope, orange curve, could be reduced by subjecting the foils to a “burn-in” procedure which we have spent significant efforts in optimizing. All optode foils ordered with multipoint calibration undergo this procedure and it should result in the aging progress shown in the blue curve.

When the luminophore in the foil is subjected to bleaching one would think that the overall decay time (lifetime) would be reduced. Due to the inverse response of the optode technology this should result in higher oxygen readings. For the oxygen optodes we see the opposite effect. The most likely explanation for this is that the luminophore in the bleaching process brakes down and forms derivate with longer lifetime.

Even though the standard oxygen foil is equipped with a black optical isolation layer some of the ambient light will penetrate the foil. This will cause similar bleaching as the excitation light. Tests suggest that the light from fluorescents indoor light affect the foils more that natural outdoor light. **For sensors in intermittent use it is therefore recommended to keep them dark at all times.** A black protection cap is available at Aanderaa and it is delivered with all new sensors.

Improvements of MkII optodes, new firmware and multipoint calibration: In the study by Takeshita et al. only 3830 optodes (Fig. 1) were used. The 3830 optodes were the first MKI version of these sensors (first delivered in 2002) and were generally only two point calibrated. The later MKII sensors (4330/4835/4831/4531, see Fig. 3) have better electronics, better optics, better temperature compensation and better formulas to calculate absolute oxygen. In addition the 4330 and 4831 optodes can be ordered with multipoint calibration (normally in 40 points at 5 temperatures and 8 oxygen concentrations) to an absolute accuracy better than ±1.5% or ±2 µM. To compensate for potential drift and temperature dependence in the electronics as well as to improve the accuracy these sensors are also equipped with a red LED that serves as a constant internal reference. Aanderaa is the only manufacturer that offers this option. A new improved firmware, called framework 3, was released in 2012 for most of the AADI smart sensors. For the 4835, 4330, 4831 and 4531 optodes these improvements include:

- Possibility to have full two-way communication with a computer through the same software, which is AADI Real Time Collector (RTC). This software can also be used to establish multisensor networks (communication with up to 60 sensors tested) and to log and display data coming from sensors in real-time.
- A more reliable “hand-shaking” and user friendly “mode settings” has been included on the serial port
- The format of the output string can be selected with a larger flexibility e.g. more text can be stripped off to save bandwidth.
- Better control of polled or non-polled mode.
- Improved data quality control has been developed e.g. information given when values are outside calibration limits.
- Use of an improved version of the Stern-Volmer equation. The improvements were suggested by Uchida et al., 2008 and are recommended by Argo float users.
- **We recommend users with custom made data loggers to update their software to take full advantage of the latest improvements. For users that do not want to update their software the sensor can be set to output data strings which are backward compatible with earlier sensor firmware versions.**
- For the oxygen optodes framework 3 has been implemented starting with the following serial numbers: 4835 s/n: 300; 4330 s/n: 1200 and 4831 s/n: 100. The 4531 optode was introduced in 2013 and is fully updated.

Fig. 3: Three versions of MKII optodes. 4835 left (300 m rated), 4330 middle (6000 m rated, 12 000 m optional) and 4831 right (6000 m)

Transparent versus non-transparent foils: The 4330 and 4831 optodes can be fitted with slower and faster responding foils. The “slower standard foils” are more robust and recommended in most applications. A black optical isolation coating protects the sensing complex from direct incoming sunlight and fluorescent particles in the water. The “faster foils” are suitable if shorter response times are required. These foils do not have any black optical isolation layer. Normally they have the same long-term stability as the “slower isolated” foils but they are noisier (resolution of ±0.2 µM). If exposed to direct sunlight they do however bleach and drift towards lower responses. It is always recommended to store sensors dark, a protecting cap is available for this purpose.

Artifacts from oxygen consumption by metal structures and sacrificial anodes: Metal structures immerged in water (of e.g. Stainless Steel, Aluminium, Bronze) are normally corrosion protected by sacrificial anodes. As the anode disintegrates oxygen is consumed at all “naked” metal parts, which the anode is in electrical contact with. The oxygen consumption can be significant e.g. during its lifetime, normally 1-2 years, a 130 g Zn anode mounted on a SeaGuard/RDCP/RCM pressure case can consume all oxygen in about 700 l of water. Water parcels with lower oxygen concentrations will form and can arrive in front of the oxygen sensors and lead to artificial dips in the oxygen readings. These effects are detectable in environments in which oxygen is stable (e.g. less than 2 % variations over time periods of days-weeks) and when currents are low (e.g. below 10 cm/s). To avoid effects of artificial oxygen consumption optodes can be cable connected, no electric contact with sacrificial anode, to SeaGuard/RDCP instruments

Artifacts from oxygen dissolution in plastic: One issue that consistently will affect the response time and accuracy of the oxygen sensors when you go through sharp oxygen gradients is how much plastic you have in your system. Plastic can dissolve considerable amounts of oxygen and it takes hours to ventilate out se Stevens, E. D. (1992) Use of plastic materials in oxygen-measuring systems. Journal of Applied Physiology, 72(2): 801-804. For profiling we believe it is unsuitable to use measuring systems with high amounts of plastic, e.g. tubes, pumps and sensors. Also Niksin bottles used for water sampling are normally made of plastic. Investigations done at cabled observatories and by mounting oxygen optodes inside Niskin bottles implies that water sampling with subsequent Winkler titration when oxygen concentrations are low (0-50 microM) consistently over-estimates the O_2 concentrations.

When oxygen goes to 0 and H_2S is produced the Winkler titrations starts to give correct values of zero O_2 again because H_2S is consuming up all the oxygen in the Niskin bottle and the readings remain 0 even when arriving on deck.

Independent investigations of AADI optode stability: AADI oxygen optodes have been used in numerous scientific studies that were published in peer-reviewed journals (see appendix 1 below for full references). Some of these studies focused on details in the performance of the AADI optodes (see references and citations below):

- **Joos et al (2003)**: *”Initial field tests have shown exceptional sensitivity and excellent stability (A. Körtzinger and D.W. R.Wallace, University of Kiel,unpublished data, 2002). The new technology seems well suited to deployment on long-term in-situ moorings, profiling floats, and other autonomous platforms.”*
- **Körtzinger et al (2004):** *”The initial results from the first 6 months of operation are presented. Data are compared with a small hydrographic oxygen survey of the*

deployment site. They are further examined for measurement quality, including precision, accuracy, and drift aspects. The first 28 profiles obtained are of high quality and show no detectable sensor drift."

- **Nicholoson et al. (2008):** *"The optode sensor showed no sign of drift when compared to Winkler measurements over the 9 months of deployment. Seaglider 021, equipped with the same optode sensor, was stable from its initial February deployment through the end of its second deployment in November, without requiring any recalibration between deployments (data not shown). The optode on glider 020 showed similar stability over its shorter deployment."*
- **Jannasch et al. (2008):** "*Oxygen optode (Aanderaa, 3930). Similar to nitrate, oxygen concentrations within estuaries can vary widely (0 to 400 µM O2). We have found the optode to be resistant to fouling as previously suggested (Tengberg et al. 2006) and to be extremely stable. Sensors were calibrated prior to deployment using the factory-suggested, two-point calibration. There was no noticeable drift in instrument accuracy before and after deployment".*
- **Hydes et al. (2009):** *"The optodes maintained good stability with no evidence of instrumental drift during the course of a year. Over the observed concentration range (230–330 mMm^{-3}) the optode data were approximately 2% low in both years. By fitting the optode data to the Winkler data the median difference between the optode and Winkler measurements is reduced to less than 1 mMm^{-3} (0.3%) in both years."* Comment: Measurements were done every 30 s. Sensors were operated 1 year at a time, which equals more than 1 Million samples.
- **Johnson et al. (2010)**: *"The oxygen sensor shows no evidence of drift, but it seems to have a small accuracy bias (≤10 µmol/l), as reported for earlier applications of Aanderaa Optode sensors on profiling floats and gliders."* The deployment period was more than 600 days.
- **Champenois and Borges (2012)** *"The comparison of O_2 measured by optodes and by Winkler titration allowed us to determine the accuracy of O_2 measurements by optodes, which was better than ±2.0 mmol kg^{-1}. The accuracy was not significantly different among the three O_2 optodes and remained stable during the study period. The precision of O_2 measurements by the O_2 optodes was better than ±0.1 mmol kg^{-1}, based upon the standard deviation on the mean of 30 measurements during 30 s, which is the standard configuration of measurements used."* Comment: The deployment period was more than 1100 days. Sensors were logged hourly which equals approximately 26,000 samples.

The longest continuous field measurements with Aanderaa optodes without detectable drift that we know of are 6 years long on Argo floats and on moored current meters.

Multipoint Calibration System: The standard calibration of the Aanderaa oxygen Optodes is based on a common characterization of a production batch of sensing foils with an additional two-point adjustment for individual Optodes and foils.
For application demanding higher accuracy (±1.5% or ±2 µM) an individual multipoint calibration is in operation since the autumn of 2012 (see Fig 4A-C below).

Fig 4A-C: A: Multipoint calibration system in operation at Aanderaa. B: Detail of multiple sensors inside the temperature regulated bath. C: Regular control of the 3 reference optodes is done with Winkler titration using an automatic titration system from SI Analytics.

The Optodes are then placed in a temperature-regulated bath where the oxygen saturation is changed by diffusing different mixtures of O_2 and N_2 into the water. The gas mixture is controlled by use of high accuracy Mass Flow Controllers. The water is stirred vigorously to provide homogeneity and oxygen concentration is referenced to three reference sensors that are calibrated with respect to high quality Winkler titrations. For continuous control water samples are also taken at regular intervals and analyzed by Winkler titration.

Aanderaa participates in an on-going calibration inter-comparison in which the performance of different leading oxygen calibration laboratories (in Australia, France, Germany, USA, Japan and Norway) is evaluated. These laboratories are mainly focusing on high accuracy calibrations of oxygen optodes for Argo floats and gliders.

For a standard multipoint calibration the oxygen is changed from 0 to 120% air saturation in 10 steps, and temperature from 1 to 30 °C in 4 steps, resulting in 40 individual calibration points. Upon customer request other calibration intervals can be performed, so far the widest interval has been between 0 to 500 % air saturation. Based on the calibration data 7 coefficients (c0 to c6) in the modified Stern-Volmer formula derived by Uchida et al, 2008 [17] are calculated:

$$[O_2] = \frac{\left(\frac{P_0}{P_c} - 1\right)}{K_{SV}}$$

and:

$$K_{SV} = c_0 + c_1 t + \ c_1 t^2$$

$$P_0 = c_3 + c_4 t$$

$$P_c = c_5 + c_6 P_r$$

where t is temperature (°C) and P_r is the raw phase shift reading (TCPhase)

After the calibration sequence the performance of all sensors are verified in 20 points covering the complete calibration range for an example coming from the standard calibration certificate see Figure 5, below.

Note that the multipoint calibration is only available for the 4330 and 4831 Optode and that in order to enable of use of the above formula the property called Enable SVUformula must be set to “yes” (requires firmware version 4.4.8 or higher).

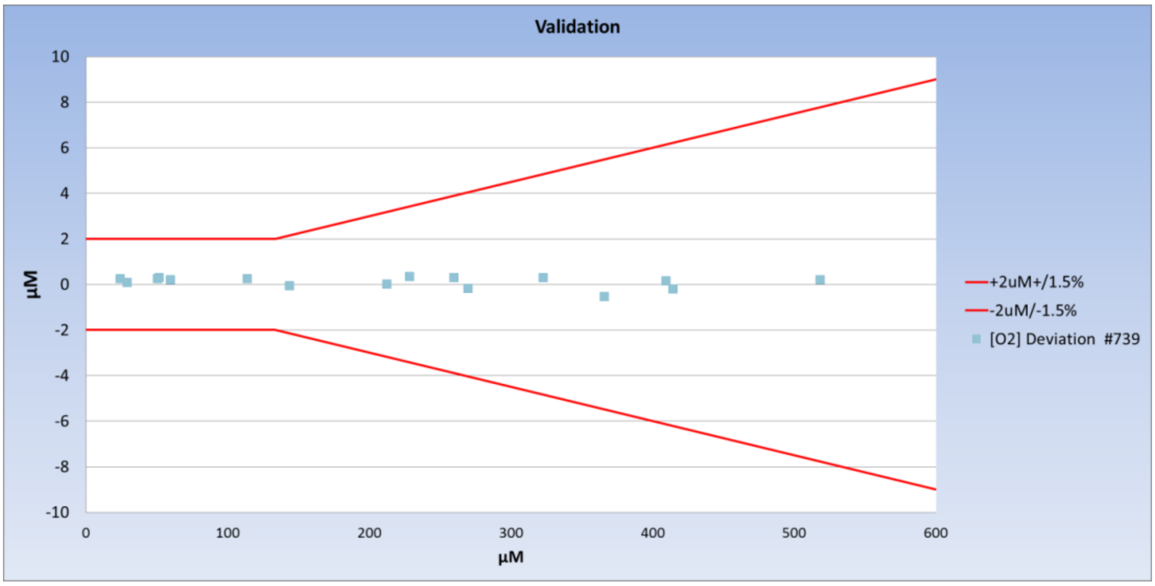

Figure 5: Typical residuals during the individual 20-point sensors check, which is performed after calibration of every multipoint calibrated oxygen optode. This type of graph is included in the individual calibration certificate delivered with each sensor

About how different manufacturers specify their sensors: Different manufacturers specify the performance of their sensors differently. It is worth taking this into account when comparing sensor specifications.

When Aanderaa states an absolute accuracy of e.g. (±1.5 % or ±2 µM) we mean the accuracy of the sensor in the field over the entire range of oxygen concentrations and temperatures. Another manufacturer might mean the laboratory accuracy just after calibration or in some cases how well the sensor returns to the exact same point as it was calibrated for right after it was calibrated. If we were to specify in this way our accuracy would be approximately ±0.5 %.

Our experience from delivering thousands of optodes over more than 10 years is that optical oxygen sensors are non-linear both in response to oxygen and to temperature. In addition their sensing foils, optics and electronics can differ. It is therefore our strong opinion that individual multipoint calibrations are necessary to achieve the highest accuracy.

When Aanderaa states a 63 % response time of 25 s for the non-transparent foil and 8 s for the faster responding transparent foil we mean the response time in water at 20°C. Other manufacturers give the response time of their sensors in air. If we were to specify in this way our 63 % response time would be approximately 6 s for the non-transparent foils and 3 s for transparent foils.

Our philosophy is to give specifications, which reflects the field performance of our sensors. We are convinced that this way of specifying is more valuable for the end user.

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Appendix 1:

The Aanderaa family of Oxygen Optodes

Common features: Extreme stability. High quality temperature sensor included. Output of calibrated values in µM and % saturation. Dynamic Range: 0-200 % (higher range at request). Calibration range: 0-120 % (higher range at request). Response time (63 % in water) standard foil t_{63}<25 s and fast foil t_{63}< 8 s (only for model 4330 and 4831). Please note that some manufacturers give response time in air, which is faster.

Model 3830: The original Aanderaa optode from 2002. 6000 m rated in Titanium. Two point calibrated: Accuracy ±5% or ±8 µM. Precision ±0.4 µM. Signal output: RS-232 and SR10 for e.g. older Aanderaa current meters. Analog output with addition of adaptor (model: 3975D).

Model 3835: 300 m rated in Plastic and Titanium. Two point calibrated: Accuracy ±5% or ±8 µM. Precision ±0.4 µM. Signal output: RS-232 and SR10. Analog output with adaptor (model: 4175C).

Model 4330/4330F (MKII, Framework 3): 6000 m rated (12 000 m optional) in Titanium. Multipoint calibrated with pre-treated foils as option: Accuracy ±1.5% or ±2 µM. Precision ±0.2 µM. Signal output: RS-232 and AiCaP (CAN bus) for e.g. Seaguard/Smartguard/Sensor strings. Fast response Temperature sensor next to foil. Red LED for better accuracy and drift control. Fast foil available (Precision ±0.4 µM).

Model 4831/4831F (MKII, Framework 3): 6000 m rated (12 000 m optional) in Titanium. Multipoint calibrated with pre-treated foils as option: Accuracy ±1.5% or ±2 µM. Precision ±0.2 µM. Signal output: RS-232 and Analog (0-5 V) for e.g. Gliders/Floats/Customer loggers. Fast response Temperature sensor next to foil. Red LED for better accuracy and drift control. Fast foil available (Precision ±0.4 µM). 8-pin Subcon connector as standard.

Model 4835 (MKII, Framework 3): 300 m rated in Plastic and Titanium. Two point calibrated: Accuracy ±5% or ±8 µM. Precision ±0.2 µM. Signal output: RS-232 and AiCaP (CAN bus). Red LED for better accuracy and drift control.

Model 4531 (Aquaoptode): 100 m rated in Plastic and Titanium. Two point calibrated: Accuracy ±5% or ±8 µM. Precision ±0.2 µM. Signal output: RS-232 and Analog (0-5 V Model 4531A, 4-20 mA Model 4531C). This sensor uses the same electronics and optics as the original sensors, with new housing and form factor price has been significantly reduced, keeping the same reliability. Red LED for better accuracy and drift control

Appendix 2:

Examples of commercially available Optode data loggers

Multiparameter
(Land/Buoy)
- Smartguard

AANDERAA

Single Channel
(0-6000 m)
- S-DOT

nke INSTRUMENTATION

Argo floats & gliders

TELEDYNE WEBB RESEARCH Everywhereyoulook

nke INSTRUMENTATION

KONGSBERG 200

develogic

Multiparameter
- SeaguardII-DCP
- Seaguard
- RDCP
- Smartsub

AANDERAA

STEINSVIK

AANDERAA

Aquaculture
- O2 cameras
- On-line systems
- Automatic Quality Control

AANDERAA

Strings
Up to 20 sensor
up to 1 km long
- SeaguardII-DCP
- Seaguard

Autonomous landers
(0-2000 m)

develogic

Appendix 3:

Some scientific papers in which Aanderaa optodes have been used and evaluated

(Last updated in April 2014)

Commercially available oxygen optodes for oceanographic application were introduced by Aanderaa in 2002. The proven long-term stability (years) and reliability of these sensors have revolutionized oxygen measurements and several thousand are in use in applications ranging from streams (Birkel et al., 2013) and buried in the river bed (Malcolm et al., 2006; 2009; 2010) to the deepest trenches (12 000 m rated) on earth, from fish farms to waste water, from polar ice to hydrothermal vents. This document gives examples of published scientific investigations in which AADI optodes have played a central role.

The basic technique and an evaluation of its functioning in aquatic environments were presented in Tengberg et al (2006). Other studies include use on autonomous floats Joos et al (2003), Körtzinger et al (2004 and 2005), Johnson et al. (2010), Alkire et al. (2012), Fiedler et al (2013), Takeshita et al. (2013), D'Asaro and McNeil (2013) and gliders (Nicholoson et al., 2008), long-term monitoring in coastal environments with high bio-fouling (Martini et al., 2007), on crab pots (Shearman and Childress, 2012), on coastal buoys (Jannasch et al., 2008; Johnson 2010; Bushinsky and Emerson, 2013), on Ferry box systems (Hydes et al., 2009), on cabled CTD instruments for profiling down to 6000 m including suggestions for improved calibrations, pressure effect and compensation for slow response (Uchida et al., 2008) and in chemical sensor networks (Johnson et al., 2007). In the Hypox project multiple optodes were used on multiple platforms to study Hypoxia (Friedrich et al., 2013). Lo Bue et al. (2011) pointed at potential artifacts in oxygen readings in environments with low currents. It has been found that the lower readings are caused by oxygen consumption occurring when metals are corroding (e.g. one sacrificial Zn anode with a weight of 130 g can consume all oxygen in 700 l of water). In Bittig et al. (2012) a seagoing multipoint Winkler free optode calibration system is described and used. McNeil and D'Asaro (2014) suggest calibration methods based on physical properties of the sensing foil. Drazen et al. (2005) presented a novel technique to measure respiration rates of deep sea fish and Sommer et al (2008) described an automatic system to regulate oxygen levels and to measure sediment-water fluxes during in-situ sediment incubation at vent sites. Wikner et al. (2013) measured respiration rates of oligotrophic waters and pointed out potential artifacts from oxygen dissolved in plastic incubators. Also Pakhomova et al (2007), Almroth et al. (2009; 2012), Viktorsson et al (2013) and Cathalot et al. (2012) used the same type of optodes on autonomous landers to perform sediment-water incubations on natural and fish farm affected sediments and with and without the introduction of sediment resuspension. In Wesslander at al (2011) the dynamics and coupling of carbon dioxide (CO_2) and oxygen was investigated in coastal Baltic Sea waters and McGillis et al (2011) described a novel method to assess the productivity of coral reefs using boundary layer and enclosure methods. Champenois and Borges (2012) studied variations in community metabolism rates of a Posidonia oceanica seagrass meadow by continuous measurements of oxygen at three different levels during three years. Viktorsson et al. (2012) used year long oxygen measurements at several Gulf of Finland locations to calibrate a 3D model for prediction of bottom water oxygen dynamics and the subsequent coupling of low oxygen conditions to release of sediment bound phosphorous. In Atamanchuk et al. (2014) pCO_2 optodes were described and used in parallel with O_2 optodes to study biogeochemical processes.

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