

INSTRUCTION MANUAL

OXY MINI Fiber Optic Oxygen Meter

Serial No._____

www.wpiinc.com

Table of Contents

1	Pref	ace	1		
2	Safe	ety Guidelines	2		
3	Des	cription of the OxyMini Device			
4	Req	uired Basic Equipment	7		
5	Plar	nar Oxygen Minisensors	8		
	5.1	Sensor Characteristic of Oxygen-Sensitive Minisensors	8		
	5.2	Housings of Oxygen-Sensitive Minisensors	12		
	5.2.1 5.2.2 5.2.3	Polymer Optical Fiber (POF) Coated with an Oxygen-Sensitive Foil Flow-Through Cell with Integrated Planar Oxygen Sensor Planar Oxygen-Sensitive Foils	12 14 15		
6	Оху	Mini Software	17		
	6.1	Software Installation and Starting the Instrument	17		
	6.2	Function and Description of the OxyMini Program	18		
	6.2.1	Menu Bar	19		
	6.2.2	Graphical Window	24		
	6.2.4	Status Bar	30		
	6.3	Subsequent Data Handling	31		
7	Cali	bration of Oxygen-Sensitive Fiber-Optic Minisensors	33		
	7.1	Calibration of a 2 mm POF Coated with a Oxygen-Sensitive Foil	33		
	7.1.1	Preparation of the Calibration Standards	33		
	7.1.2	Mounting the Oxygen-Sensitive Minisensors	34		
	7.1.3	Calibration without Automatic Temperature Compensation	34		
	7.1.4	Manual Calibration	37		
	7.2	Calibration of Flow-Through Cell with Integrated Oxygen Sensor	42		
	7.2.1	Preparation of the Calibration Standards	42		
	7.2.2	Mounting the Flow-Through Cell Oxygen Sensors	43		
	7.2.3	Calibration without Automatic Temperature Compensation	44		
	7.2.4	Calibration with Automatic Temperature Compensation	48		
	7.3	Calibration Planar Oxygen-Sensitive Foils Integrated in Glass Vials	53		
	731	Preparation of the Calibration Standards	53		
	7.3.2	Mounting Planar Oxygen-Sensitive Foils	54		
	7.3.3	Calibration without Automatic Temperature Compensation	54		
	7.3.4	Calibration with Automatic Temperature Compensation	57		
Q	7.3.5 Moa	Manual Calibration	86		
U	0 1	Magouroment with DOEs Costed with an Owner Sensitive Est	00		
	0.1	Measurement with a Flow Through Callwith Intermeted Owners Concern	00		
	ð.2	measurement with a Flow-I nrough Cell with Integrated Oxygen Sensor	60		
	0 9				

8.4	Some Advice for Correct Measurement	62
8.4.1	Signal drifts due to oxygen gradients	62
8.4.2	Signal drifts due to temperature gradients	62
8.4.3	Signal drift due to photo-decomposition	
0.4.4	Signal difficulte to too much ambient light	
8.4.6	Correction for air-pressure variations	64
9 Ger	neral Instructions:	65
9.1	Warm-Up Time	65
9.2	Maintenance	65
9.3	Service	65
10 Tec	hnical Data	66
10.1	General Data	66
10.2	Analog Output and External Trigger	68
10.3	Technical Notes	69
10.4	Operation Notes	69
11 Iro	uble Shooting	70
12 App	pendix	71
12.1	Basics in Optical Sensing of Oxygen	71
12.1.	1 Dynamic Quenching of Luminescence	71
12.1.	2 Major Components of Fiber-Optic Minisensors	
12.1.	Advantages of Optical Oxygen-Sensitive Minisensors	
12.1.	5 LiteratureError! Bookmark not	defined.
12.2	Determination of the Oxygen Concentration Using a Modified Stern-Vo	Imer
	Equation	
12.3	Oxygen Conversion Formulas	76
12.4	Temperature Dependent Constants Affecting the Oxygen Content	78
12.4. 12.4.	 Water Vapor Pressure	78 79
12.4.	3 Dependence on the Salt Concentration	83
12.5	Temperature Compensation of the Response of Oxygen Sensors	84

1 Preface

Congratulations!

You have chosen a new innovative technology for measuring oxygen!

The OxyMini is a compact, easy to transport and completely PC-controlled fiber optic oxygen meter. The data evaluation is PC supported as well.

The OxyMini was specially developed for small fiber optic oxygen sensors, flow trough cells and integrated sensor systems. It is based on a novel technology, which creates very stable, internal referenced measured values. This enables a more flexible use of oxygen sensors in many different fields of interest.

Optical oxygen sensors (also called optodes) have important advantages over common Clark type electrodes:

- They are small
- They do not consume oxygen
- Their signal does not depend on the flow rate of the sample
- They have excellent long term stability
- They can be physically divided from the measuring system which means a contactless measurement.
- They can be autoclaved and γ-sterilized as well

Therefore, they are ideally suited for the examination of small sample volumes, long term measurements in difficult samples, and for biotechnological applications.

A set of different oxygen minisensors, flow through cells and integrated sensor systems is available to make sure you have the sensor which is ideally suited to your application.

Please feel free to contact our service team to find the best solution for your application.

2 Safety Guidelines

PLEASE READ THESE INSTRUCTIONS CAREFULLY BEFORE WORKING WITH THIS INSTRUMENT!

This device has left our works after careful testing of all functions and safety requirements.

The perfect functioning and operational safety of the instrument can only be ensured if the user observes the usual safety precautions as well as the specific safety guidelines stated in these operating guidelines.

- Before connecting the device to the electrical supply network, please ensure that the operating voltage stated on the power supply corresponds to the mains voltage
- The perfect functioning and operational safety of the instrument can only be maintained under the climatic conditions specified in Chapter 11 "Technical Data" in this operating manual.
- If the instrument is moved from cold to warm surroundings, condensate may form and interfere with the functioning of the instrument. In this event, wait until the temperature of the instrument reaches room temperature before putting the instrument back into operation.
- Balancing, maintenance and repair work must only be carried out by a suitable qualified technician, trained by us.
- Especially in the case of any damage to current-carrying parts, such as the power supply cable or the power supply itself, the device must be taken out of operation and protected against being put back into operation.
- If there is any reason to assume that the instrument can no longer be employed without a risk, it must be set aside and appropriately marked to prevent further use.
- The safety of the user may be endangered, e.g., if the instrument
 - is visibly damaged;
 - no longer operates as specified;
 - has been stored under adverse conditions for a lengthy period of time;
 - has been damaged in transport
- If you are in doubt, the instrument should be sent back to the manufacturer WPI for repair and maintenance.
- The operator of this measuring instrument must ensure that the following laws and guidelines are observed when using dangerous substances:
 - EEC directives for protective labor legislation;
 - National protective labor legislation;
 - Safety regulations for accident prevention;
 - Safety data-sheets of the chemical manufacturer

The OxyMini is not protected against water spray; The OxyMini is not water proof; The OxyMini must not be used under environmental conditions, which cause watercondensation in the housing; The OxyMini is sealed; The OxyMini must not be opened;

We explicitly draw your attention to the fact that any damage of the manufacturer seal will render of all guarantee warranties invalid.

Any internal operations on the unit must be carried out by personal explicitly authorized by WPI and under antistatic conditions.

The OxyMini may only be operated by qualified personal.

This measuring instrument was developed for use in the laboratory. Thus, we must assume that, as a result of their professional training and experience, the operators will know the necessary safety precautions to take when handling chemicals.

Keep the OxyMini and the equipment such as TEMP 100 temperature sensor, power supply and optical sensors out of the reach of children!

As the manufacturer of the OxyMini, we only consider ourselves responsible for safety and performance of the device if

- the device is strictly used according to the instruction manual and the safety guidelines
- the electrical installation of the respective room corresponds to the DIN IEC/VDE standards.

The OxyMini and the sensors must not be used in vivo examinations on humans!

The OxyMini and the sensors must not be used for human-diagnostic or therapeutical purposes!

3 Description of the OxyMini Device

The **OxyMini** is a precision, *temperature compensated*, oxygen meter, designed for fiber-optic oxygen minisensors.

Their robust design and low power consumption makes it ready for indoor and **outdoor** application.

For operation, a PC/Notebook with RS232 interface is required. The OxyMini is controlled using a comfortable software, which also saves and visualizes the measured values



OxyMini instrument features:

- high precision
- portable (battery power optional)
- analog/digital data output (on request)
- temperature compensation

There also exits the possibility to combine several single OxyMini oxygen meter to obtain a multichannel system. It allows the user to create a 2, 3, 4 or more channel system.



The OxyMini oxygen meter contains a dual 12 bit analog output and an external trigger input. The analog output values can be programmed with the PC software (included). The user can choose between oxygen, temperature, amplitude or phase for each channel independently. The OxyMini can be used as a stand-alone instrument when combined with an external data logger.

Front Panel



ELEMENT	DESCRIPTION	FUNCTION		
S1	ON/OFF switch	Switches the device ON and OFF		
C1	SMA fiber connector	Connect the fiber optic oxygen minisensor here.		
L1	Control LED	red: instrument off; green: instrument on; orange: stand by;		
T1	Connector for TEMP 100 temperature sensor	Connect the TEMP 100 temperature sensor for temperature compensated measurements here.		

Rear Panel of the OxyMini device

Two standard BNC connectors are added for analog output channels 1 and 2, another one for external trigger input. See figure below.

The electrical specifications of all rear panel connectors are given in technical specification sheet. Please read also the technical notes to avoid mistakes.



The rear panel of *OxyMini-AOT* differs from *OxyMini*. Here two standard BNC connectors are added for analog output channels 1 and 2. See figure below.

The electrical specification of all rear panel connectors is given in the technical specification sheet. Please read also the technical notes to avoid mistakes.

ELEMENT	DESCRPTION	FUNCTION
C2	Line adapter for power supply	Connector for 9 - 36 V DC power supply.
C3	RS232 interface (male)	Connect the device with a RS232 data cable to your PC/Notebook here.
A1	Analog out (channel 1)	Connect the device with external devices, e.g. a data logger
A2	Analog out (channel 2)	Connect the device with external devices, e.g. a data logger
T1	External trigger input	Connect the device with external devices, e.g. data logger with a trigger output, pulse generator

Features

- minisensor oxygen meter with temperature compensation
- 2 x 12bit, programmable analog channels, with optical isolation
- 9 36 V supply voltage (or 220/110V AC adaptor)
- RS 232 interface, with optical isolation
- robust metal box

4 Required Basic Equipment

- Oxygen meter OxyMini*
- Software for OxyMini*
- PC / Notebook (System requirements: Windows 95/98/2000/XP//Millenium/NT 4.0; Pentium processor, at least 133 MHz, 16 MB RAM)
- RS 232 Cable *
- Line adapter (110 220 V AC, 12 V DC) *
- Temperature sensor TEMP 100*
- Oxygen-sensitive minisensor
 The minisensors are mounted into different types of housings
- Vessels for calibration standard 100 (water-vapor saturated air = 100 % air saturation) and calibration solution 0 (oxygen-free water)
- Laboratory support with clamp, micro-manipulator
- *: scope of supply

5 **Planar Oxygen Minisensors**

5.1 Sensor Characteristic of Oxygen-Sensitive Minisensors

The principle of the sensor operation is based on the quenching of luminescence caused by collision between molecular oxygen and luminescent dye molecules in the excited state. Figure 5.1 shows a typical response curve of an oxygen-sensitive sensor. In the presence of oxygen the signal - in our case the phase angle Φ - decreases. The phase angle Φ can be related to the oxygen content as shown in Figure 5.2. The theoretical aspects are explained more detailed in the appendix.



the oxygen concentration.

different oxygen contents

Measuring range

The optimal and maximal measuring ranges of WPI sensor membranes is given in Table 5.1, which displays the measuring range of WPI oxygen sensors in different oxygen units.

Table 5.1 Measuring range of WPI oxygen sensors.

oxygen unit	optimal measuring range	maximal measuring range*			
oxygen sensor					
air-saturation	0 - 250 %	0 - 500 %			
oxygen-saturation	0 - 50 %	0 - 100 %			
hPa	0 - 500 hPa	0 - 1000 hPa			
Torr	0 - 400 Torr	0 - 760 Torr			
mg/L (≡ppm)	0 - 22.6 mg/L (≡ppm)	0 - 45 mg/L (≡ppm)			
μmol	0 - 800 µmol	0 - 1.4 mmol			

*Please contact the WPI technical support group to get the appropriate software for sensor membrane when measuring up to 100 % oxygen.

Resolution

The WPI OxyMini has a phase resolution of smaller than 0.05°. Since the oxygen calibration plot displays a non-linear behavior, the oxygen resolution is given for three different partial pressures at 20 °C. The resolution in oxygen is also transformed in different oxygen units.

Table 5.2.	Oxygen	resolution	at different	oxygen	contents a	t 20 °C	and 101	3 mbar.
				<i>si</i> , <i>jssi</i> .				• • • • • • • • •

Sensor membrane

hPa	% air-sat.	<i>mg/L</i> (≡ppm)	
2 ± 0.1 hPa	1 ± 0.05 % air-sat.	0.09 ± 0.005 mg/L	
$60\pm0.3~\mathrm{hPa}$	30 ± 0.1 % air-sat.	2.72 ± 0.01 mg/L	
$200 \pm 1 \text{ hPa}$	100 \pm 0.5 % air-sat.	9.06 ± 0.05 mg/L	
500 ± 0.3 hPa	250 \pm 1.7 % air-sat.	$22.65\pm0.15~\text{mg/L}$	
Torr	% oxygen	µmol	
$1.55\pm0.08~\text{Torr}$	0.21 ± 0.01 % oxygen	2.83 ± 0.14 µmol	
46.7 ± 0.2 Torr	6.3 ± 0.02 % oxygen	85.0 ± 0.28 µmol	
$155.5\pm0.75~\text{Torr}$	20.9 ± 0.1 % oxygen	283.1 ± 1.4 µmol	
388.8 ± 2.6 Torr	52.4 ± 0.35 % oxygen	798.0 ± 4.7 µmol	

Temperature

WPI oxygen sensors can be used in the temperature range of -10 to 50 °C. WPI offers a TEMP 100 temperature sensor in combination with the OxyMini to record temperature variations which are compensated using the OxyMini software (see Chapter 7&8, *Calibration of Oxygen Sensors* and Chapter 9, *Measurement*). In the appendix you will find a detailed description of the *Temperature Dependent Constants Affecting the Oxygen Content* (chapter 14.4) and *Temperature Compensation of the Response of Oxygen Sensor* (chapter 14.5).

Cross sensitivity:

There exists no cross sensitivity for carbon dioxide (CO₂), hydrogen sulfide (H₂S), ammonia (NH₃), pH, any ionic species like sulfide (S₂⁻), sulfate (SO₄²⁻) or chloride (Cl⁻).

The sensors can also be used in methanol- and ethanol-water mixtures as well as in pure methanol and ethanol.

We recommend avoiding other organic solvents, such as acetone, chloroform or methylene chloride, which may swell the sensor matrix.

Interferences were found for gaseous sulfur dioxide (SO_2) and gaseous chlorine (Cl_2) . Both of them lead to too high oxygen concentrations.

Response time

The response time (t_{90}) of the oxygen sensor is dependent from the diffusion rate of oxygen through the sensor layer, hence, the response time is dependent from the thickness of the sensor layer and the stirring rate. A typical oxygen response curve of sensor membrane in a non-stirred and stirred sample solution is shown in Figure 5.3. The response times (t_{90}) of sensor membranes is listed in Table 5.3.

Unlike electrodes, optical sensors will **not** consume oxygen and the signal is independent of changes in flow velocity which means that stirring decreases the response time, but has no effect on the measured value.

Optical isolation of the oxygen-sensitive layer which is applied to exclude ambient light and improve chemical resistance will slow down the sensor response.

Figure 5.3 Response characteristic of an optical isolated oxygen sensor in a stirred, a non-stirred sample solution and in the gas phase.

	dissolved oxygen		gaseous oxygen
	stirred	not stirred	
oxygen sensor			
t90 without optical isolation	< 35 s	< 50 s	< 8 s
t_{90} with optical isolation	< 40 s	< 60 s	< 10 s

Table 5.3 Response time (t_{90}) of WPI oxygen sensors.

Optical isolation

Optical isolated sensor tips are required, if your sample shows intrinsic fluorescence between 540 - 660 nm. Consequently, an optical isolation is recommended measuring in whole blood, urine or chlorophyll containing samples. Using optical isolated sensors exclude the impact of colored samples and ambient light on measurements. Furthermore, the optical isolation layer improves chemical resistance of the sensor membranes.

Optical isolated sensor tips of oxygen sensors enable measurement in photosynthetically active samples, since stimulation of photosynthesis, due to emission of blue-green light from the fiber tip, is avoided.

WPI offers additional optical isolation for all types of oxygen sensors. However, using opticalisolated sensor tips, the layer thickness increases. Since the response time of the sensor is dependent from the diffusion rate of oxygen through the sensor layer, additional optical isolation, of course, increases the response time (t_{90}). The response time of oxygen sensor with and without optical isolation are compared in Table 5.3.

Ambient light

The sensor should be shielded from ambient light to obtain reliable data. A black overcoat, called optical isolation is strongly recommended to reduce ambient light.

Photo-Decomposition

The oxygen-sensitive material may be subject to photo-decomposition resulting in a signal drift. Photo-decomposition takes place only during illumination of the sensor tip and depends on the intensity of the excitation light.

Drift in % air-saturation at 100% air-saturation when illuminating the oxygen sensor for 1, 12 and 24 hours in the continuous mode.

	Drift per hour	Drift per 12 hours	Drift per 24 hours
Oxygen minisensor			< 0.4 % air-saturation

5.2 Housings of Oxygen-Sensitive Minisensors

WPI fiber-optic oxygen sensors are based on 2 mm polymer optical fibers (POF). Depending on the respective application, WPI offers a set of different standard designs.



2 mm **POF** with coated sensor Flow foil connected sensor

Flow through cell design planar connected to a 2 mm POF foils

design planar oxygen sensitive sensor

Of course, it is possible to build *customer-specific* designs. Please feel free to contact our service team to find the best solution for your application.

5.2.1 Polymer Optical Fiber (POF) Coated with an Oxygen-Sensitive Foil



This oxygen sensor consists of a polymer optical fiber (POF) with a polished distal tip which is coated with a planar oxygensensitive foil.

The end of the polymer optical fiber is covered with a high-grade steel tube, to protect both the sensor material and the POF.

Usually, the fiber is coated with an optical isolated sensor material in order to exclude ambient light from the fiber tip.

Schematic drawing

Features

- usable for process application
- very robust sensor with an excellent long-term stability (more than 100000 data points without drift)
- sterilizable (H₂O₂, EtOH)
- not autoclavable (POF does not stand autoclaving conditions (130 °C, 1.5 atm) Sensor coating is autoclavable; for autoclavable sensors please refer to our oxygen sensor foils.)
- Shelf-life: This oxygen sensor has a long lifetime. If the oxygen-sensitive foil delaminates from the POF you can send the fiber back to WPI for re-coating service.

Ordering information

WPI # 501641MiniTip, fiber-optic oxygen sensor (2.3 mm OD)Wide-range membrane (0-100% pure oxygen)The standard cable length is 2.5 m.

5.2.2 Flow-Through Cell with Integrated Planar Oxygen Sensor



WPI offers **very robust** gas- and pressure tight (up to 2 bar) miniaturized fiber-optic flow-through cells MiniFlow with integrated oxygen sensors. The flow-through cell is connected to the WPI oxygen meter OxyMini using polymer optical fibers (POF) with 2 mm diameter as a light guide. Inlet and outlet tubes of the flow-through cell probe can be connected via Luer-Lock adapters. Liquids (water, blood) can be pumped through the cell. Online monitoring is possible.

Schematic drawing of flow-through cell oxygen sensors

Features

- very robust sensor with an excellent long-term stability (more than 100000 data points without drift)
- online monitoring
- sterilizable (autoclave (130 °C, 1.5 atm), EtOH, H₂O₂)
- response time (t₉₀) in the order of 1 minute

Ordering Information

WPI # 501642 MiniFlow, flow-through cell with integrated planar oxygen sensor
 Wide-range membrane (0-100% pure oxygen)
 The standard cable length is 2.5 m.

5.2.3 Planar Oxygen-Sensitive Foils



Planar oxygen sensors immobilized onto a different support (polyester, glass) are available for customer specific applications. Sensors based on a polyester support can be easily cut into small pieces using a razor blade. Round spots (sensor spots) of 3 mm in diameter can be punched.

The sensor spots can be glued, for example, inside glass vials (e.g. cell culture flask) and the oxygen concentration can be measured non-invasive and nondestructive from outside through the wall of the flask.

Prerequisite: The wall of the flask has to be transparent and non fluorescent

Features

- non-invasive and non-destructive measurement from outside through the wall of the flask
- excellent mechanical stability and long-term stability (more than 100000 data points without drift)
- online monitoring
- response time (t₉₀) in the order of 40 s
 - Oxygen sensor immobilized onto a glass support
 - stands CIP (Cleaning In Place) conditions
 - sterilizable (autoclave (130 °C, 1.5 atm), EtOH, H₂O₂)

Oxygen sensor immobilized onto a polyester support

- stands CIP (Cleaning In Place) conditions
- sterilizable (EtOH, H₂O₂)
- not autoclavable
- flexible



A polymer optical fiber is used as a light guide between the OxyMini oxygenmeter and a sensor foil MiniFoil which was glued inside a glass vial to read out the analyte concentration non-invasive and non-destructive **from outside through the transparent wall of the flask**.

Ordering information

WPI # 501643	MiniFoil, planar oxygen-sensitive foil		
	Wide-range membrane (0-100% pure oxygen)		
	The dimensions of oxygen sensor are 1 cm by 1 cm.		

6 OxyMini Software

This software is compatible with Windows 95/98/2000/Millenium/NT4.0/XP.

6.1 Software Installation and Starting the Instrument

- Insert the supplied disc/CD into the respective drive. Copy the file oxymini01.exe onto your hard disk. (for example, create C:\OxyMini\oxymini01.exe). Additionally, you may create a link (Icon) on your desktop.
- 2. Connect the OxyMini via the supplied serial cable to a serial port of your computer. Tighten the cable with the screws on your computer and on the OxyMini.
- 3. Connect the power supply.
- 4. Please close all other applications as they may interfere with the software. Start the program oxymini01.exe with a double click. The following information window appears:

Connect the instrument to the \ensuremath{PC} .			
waiting			

5. If the right com port is adjusted this information window disappears within a few seconds. If the wrong com port is adjusted you are asked to set the right com port:

Connect the instrument to the PC .
waiting
And choose the right com port.
Com Port

With a left mouse click onto '*com port*' you are able to set the right com port. Please confirm your selection by clicking the '*OK*' button. The information window disappears if the right com port is adjusted.

Select COM Port	
Com 1	
√ <u>o</u> k	X Cancel

6.2 Function and Description of the OxyMini Program

The window shown below is displayed after starting the software microx.exe: The program has 4 main sections:

- 1. Menu bar
- 2. Graphical window
- 3. Status bar
- 4. Control bar, divided into numerical display, control buttons and warning lights



6.2.1 Menu Bar

File	Charts	Display	
→ Exit	→ Oxygen	→ Zoom	
			→ AutoScaleY1
			→ Undo Zoom
	→ Phase		
	→ Amplitude	→ Clear Charts	
	→ Temperature	→ Dimensions	

Print	Settings
→ Charts	→ Com Port
	→ Instrument Info
	→ analog settings
	\rightarrow LED Intensity

File

Exit

Closes the program.

Charts

The respective charts of the measurement can be displayed ($\sqrt{}$) or hidden

Oxygen:

Oxygen content in the chosen unit

Phase:

Phase angle, the raw data

Amplitude:

The magnitude of the sensor signal

Temperature:

The measured temperature

Display

Zoom:

<u>F</u> ile	<u>C</u> harts	<u>D</u> isplay	<u>P</u> rint	<u>S</u> ettii	ngs			
		Zoon	n –	•	~	<u>A</u> utoSca	aleY1	
		<u>C</u> lear	Charts	s		<u>U</u> ndo Zo	oom	
	0.	Djme	nsions		Ŀ.	▼		Qui

AutoScaleY1 is the default setting. AutoScaleY1 means that the y-axis is scaled

automatically.

Undo Zoom: The original display is recovered; see also graphical display

Clear Charts: The graphs shown on the display is cleared.

Dimensions:

Dimension Settings	You can adjust the number of
Choose the dimensions for the chart:	measurements points on the x-axis shown in the display (maximum number of points are 5000)
X-axis (ticks)	Furthermore, you can adjust the minimum and the maximum of the y-axis.
Y-axis (minimum) 🛛 🗧	The AutoScaleY1 function is switched
Y-axis (maximum) 🛛 🚺	off.

Print

Charts: The charts shown in the display can be printed

Settings

ComPort

The serial com-port (com1 – com2) for the serial interface (RS 232) can be chosen in this window. COM 1 is the default setting. If you choose the wrong Com port, the information window '*Connect the instrument to the PC and choose the right com-port*' does not disappear.

Instrument Info:

Here you can find the version of the software and some important settings of the instrument. If you have a problem with the OxyMini oxygen meter, please contact our service team and have the software and instrument information ready.

To change back to the graphical window click the 'Measure Chart' button.

Instrument Info

MEASURE CHART INFO

IDENTIFICATION PHIboard number : 20020007 PM number : 20020043 Serial number : FB3-opt01-AOT-03-001 MUX channel : OFF - 00

PARAMETERS Signal LED current: 030 Ref LED current : 170 Ref LED amplitude : 95916 Frequency : 005 Sending interval : 0001 Averaging : 1

SYSTEM SETTINGS APL function : ON Temp compensation : ON - ch a Analog out : chA o chB t RS232 echo : OFF Data logger : OFF - 0/800 Oxygen unit : %a.s.

🖺 Print Info

٠

-

LED-Intensity

With the current of the LED you can adjust the amount of light illuminating the sensor spot.

You can choose between an '**Auto Adjust**' of the LED where the OxyMini adjusts the optimal LED current itself, or you can select '**Advanced**' where you can adjust the LED current yourself.

If you increase the LED current, the signal amplitude increases, since a higher light density illuminates the sensor spot.

Auto Adjust:

To make the adjustment of the LED intensity automatically, just click the button '**Start Auto Adjust**'. Please check that the oxygen microsensors has been connected to the instrument.

LED Intensity Adjust		
Auto Adjust Advanced		
Start Auto Adjust		
Status :		
<u>✓ C</u> lose		

The automatically adjustment of the LED intensity is finished when in the status window the message '**Auto adjustment finished**' appears. Click the '**Close**' button to confirm the settings.

LED Intensity Adjust		
Auto Adjust Advanced		
Start Auto Adjust		
Status : Auto adjustment finished.		
<u>✓ C</u> lose		

Advanced:

Click the '**Advanced**' button to change the LED current manually. Values between 10 and 100 % are possible. After clicking the 'confirm' button you can see the change of the amplitude in the window below.

LED Intensity Adjust		
Auto Adjust Advanced LED Intensity 45 🚔 % 🗸 Confirm		
amplitude 11092		
<u>✓ C</u> lose		

Please note, that after changing the LED intensity you should re-calibrate the oxygen microsensor. A warning window points you to re-calibrate the oxygen microsensor.

LED Adjust - Wa	rning		×
The calibration values are no longer valid.			
Please recalibrate the sensor.			
	🗸 Calibration	🗙 I will recalibrate later.	

Please note:

By increasing the light intensity you increase the amplitude of the oxygen microsensor. This leads to smoother phase signals. However, increasing the light intensity can increase photobleaching, which decreases the shelf-life of your sensor.

Analogue output

Here you can choose which data are exported via the analog output. The OxyMini device has two analog outputs and one trigger input. The desired data sources (oxygen, temperature, amplitude, phase) can be chosen via the dialog box.

Equivalence coefficient

oxygen	1 : 0.1 (e.g. 973mV = 97.3 % air saturation)
temperature	1 : 0.1 (e.g. 208mV = 20.8°C)
amplitude	1 : 20 (e.g. 1110mV = 22200 relative units)
phase	1 : 0.025 (e.g. 1100mV = 27.50°)

Analog Choice			
Please define the analog output ports.			
analog channel 1:	analog channel 2:		
O - none	C - none		
💿 - oxygen (airsat.)	C - oxygen (airsat.)		
C - phase	C - phase		
C - amplitude	C - amplitude		
C - temperature	 temperature 		
	¥ Cancel		

Please note:

If you have adjust the desired settings of the analog outputs and want to connect the instrument to a datalogger please close the software to store the settings before you disconnect the OxyMini from the computer.

6.2.2 Control Bar

Numerical display



The actual oxygen content in the chosen unit (here % air-saturation) is displayed in the oxygen window. The oxygen unit can be changed by clicking the pull down menu. Tables and formulas for the calculation of different concentration scales are given in the appendix.

Please note:

It is also possible to change the oxygen unit during the measurement.

Temperature measurement:

The actual temperature value of the sample (in the case of temperature compensated measurements) is displayed in the temperature window.

If measurement is performed without temperature compensation, the manual inserted temperature is displayed with the hint that temperature measurement is off-line.

Control buttons:

The way to start a measurement is

- (A) Calibration of the minisensor with the Calibration Assistant
- (B) Start Measurement with Assistant
- (C) Log Data

Measurement Calibration			
Quick Start	Advanced Start	Stop	
Sampling Rate :	1 sec		
Log Data			

(A) Calibration:

The calibration assistant is opened (see chapter 7, Calibration of Oxygen-Sensitive Minisensors);

(B) Measurement:

The measurement assistant is opened (default setting).

Quick Start:

The measurement is started. The measurement settings are continuous mode which means that each second a new measurement data is recorded. The measurement is temperature compensated i.e. a temperature sensor has to be connected. If no temperature sensor is connected the following warning window appears.

Warning. No temperature sensor detected. Please check the connection.
Close

Click the '**Close**' button if you want to continue the measurement without temperature compensation. The temperature is set to 20 °C by the software and temperature compensation of the oxygen content is based on 20 °C.

Connect the temperature sensor if you want to perform a temperature compensated measurement.

If you want to change the measurement stetting click the 'Advanced start' button.

Please note:

The measurement values are not stored. Click the 'Log Data' button to store the measurement data.

Advanced Start:

In the 'Advanced Start' mode it is possible to adjust user-defined measurement settings.

If you haven't performed sensor calibration yet the following window appears

Measurement Assistent	- D ×
You did not calibrate the sensor after program start.	
Date of last calibration : 28/11/02 Last calibration was made 7 days ago.	
New Calibration	
<u>Continue</u> <u>X</u> <u>C</u> ancel	

If you want to measure with the last sensor calibration - you can find the '*date of the last calibration*' in the window - click the '*Continue*' button. To obtain reliable results we strongly recommend to perform a sensor calibration before measurement by clicking the '*New calibration*' item when connecting a new sensor.

Follow the instructions given in chapter 7 to calibrate the respective minisensor.

To leave this menu click the 'Cancel' button.

If you have already performed sensor calibration, the measuring assistant will be opened.

In this window you can choose the measurement settings:

Measurement Assistent	
Choose the measurement setti	ngs:
Sampling Rate:	
1 sec	
T	
I emperature Compensation	
⊙ off	Please enter the temperature for measurement:
• on	<u>►</u> 20_0 <u>►</u> ℃ <u>✓ Start</u>
<u>✓ S</u> tart	X Cancel

In the 'Sampling Rate' window you can select the desired measurement mode with a dropdown menu.

Choose sampling rate	:
1 sec 💌	
fast sampling]
1 sec	
_ 5 sec	
10 sec	ation
30 sec	
¶ 1 min	
5 min	
10 min	
d 30 min	
60 min	

By clicking the drop down menu you can choose from '*fast sampling*' (update rate each 250 – 350 ms) to the '*60 min*' mode where each hour a measuring point is recorded.

The speed of recording a measurement point in the '**fast sampling**' mode is about 250 ms when no temperature sensor is connected and decreases to about 350 ms when connecting a temperature sensor or activating the analog output channels.

Please note:

The sensor shelf life can be increased using a slower measuring mode since the effect of photo-bleaching is reduced. The illumination light is switched off between sampling. A further advantage using a high measuring mode is that huge amounts of data for long-time measurement can be avoided.

In the '*temperature compensation*' window you can decide whether you want to measure with or without temperature compensation.

If you want to measure with temperature compensation, click the '**ON**' button. Please ensure that the temperature sensor TEMP 100 is connected to the OxyMini, before you click the '**Start**' button to continue. The window where you can enter the temperature manually is disabled.

Temperature Compensation • on • off	Please enter the temperature for measurement:
<u>✓ S</u> tart	X Cancel

If you want to measure without temperature compensation, choose the '**OFF**' button. You will now be requested to enter the temperature of the sample manually. Click the '**Start**' button to start the measurement.

Temperature Compensation	
⊖ on	Please enter the temperature for measurement:
☞ off	<u>▼</u> 20 0 ▼ °C
<u>√ S</u> tart	X <u>C</u> ancel

(C) Log Data:

To store the data of your measurement click the '*Log Data*' item. Next to the '*Log Data*' item an information window displays whether the actual measurement is stored to a file (*logging*) or not (*no logging*);

Log Data	no logging	

The measurement description which you can enter in the text field '*Enter a description to the header of the file*' is stored in the Ascii File.

By clicking the button '*Choose File*', you can select the location where you want to store the data. Choose as file extension **.txt*. Click the '*Save*' button to confirm your settings.

File Description and Logging	<u> </u>
[Enter a description to the header of the file.]	
Choose File	
🗙 <u>C</u> ancel	

Data file sele	ection				? ×
Speichern jn:	≙ Eigene Dateien	-	E	1 🖻	
Adobe					
L					
Datei <u>n</u> ame:	test measurement			<u>S</u> pe	ichern
Datei <u>t</u> yp:	Data Files (*.txt)		•	Abbr	echen

By clicking the '**Stop Log Data**' item you stop data logging which is displayed by the blinking '**no logging**' in the information window next to it.

Stop Log Data	logging	

Stop Measurement

The measurement is ended by a left click on the '*stop*' button in the control bar.

Warning Lights:

At the right bottom of the window you can find the amplitude, phase angle and three warning lights. The warning lights are explained below:

٢	amplitude	
۲	phase	
۲	ambient light	
	Display Raw Values	

amplitude:	red:	Amplitude is too low, the sensor tip may be damaged or sensor cable may not be connected
	yellow:	Amplitude is critically low, replacement of the sensor is recommended
	green:	amplitude is correct
phase:	red: green:	phase angle is out of limits phase angle is in normal range
ambient light:	red:	background light (e.g. direct sunlight, lamp) is too high. Decrease of false light is recommended
	green:	ratio of sensor signal to faise light is acceptable

By clicking the 'Display Raw Values' button, the raw data of phase angle and amplitude are displayed next to the warning lights.



6.2.3 Graphical Window

The respective sensor signal is displayed according to the selection of the 4 control buttons oxygen, phase, amplitude and temperature (menu chart). The oxygen content is displayed in the chosen unit, the temperature in °C. The raw values (the phase angle in degrees and the sensor amplitude in mV) can also be displayed by clicking the button **'Display Raw values'**.

Zoom Function:

- 1. Press the left mouse button and drag from left to right to enlarge a certain area of the graphical window. The graphical window displays the selected data points and is not actualized with new data.
- 2. Press the left mouse button and drag from right to left to recover the original display, or click the '**Undo Zoom**' button in the *display* menu under *zoom*.

6.2.4 Status Bar



- **sw1:** Displays the serial port which is used for communication of the OxyMini device with the PC
- **sw2:** Displays the file name in which the measurement data are stored. "No storage file selected" is displayed if no file was selected (no data storage).
- sw3: Displays the start time of the measurement
- **sw4:** Displays the actual time

6.3 Subsequent Data Handling

In the head of the ASCII file, you find the **description** of your measurement which you have entered by storing the file.

Below you find the '*instrument info*' containing the data of the complete calibration routine and some more important settings of the instrument and firmware.

The '**software info**' below contains the version number of the OxyMini software, date and time of the performed measurement. If there is a problem with the OxyMini oxygen meter, please contact our service team and have the software and instrument information ready.

Below, you find the '*measure mode settings*' containing the *dynamic averaging*, and the *measuring mode*.

The following rows, separated by semicolons, list the measuring data. The first two rows contain the **date** and **time**, the third the **log-time** in minutes, the fourth the **oxygen content** in the chosen unit. The raw data - phase **angle** in [°] and the **amplitude** in [mV] - are stored in the fifth and sixth row, respectively. The seventh row contains the **temperature** in °C measured by TEMP100 temperature sensor. Raw data can be used for user defined recalculations according to the formulas and tables listed in the appendix.

***** DESCRIPTION ********* Instrument check ***** INSTRUMENT INFO ******* *** IDENTIFICATION PHIboard number : 20020007 PM number : 20020043 Serial number : AOT-03-001 MUX channel : OFF - 00 PARAMETERS Signal LED current: 030 Ref LED current : 170 Ref LED amplitude : 95916 : 005 Frequency Sending interval : 0001 Averaging : 1 SYSTEM SETTINGS APL function : ON Temp compensation : ON - ch a Analog out : chA o chB t RS232 echo : OFF Data logger : OFF - 0/800 Oxygen unit : %a.s. CALIBRATION Sensor type : 2 0% a.s. phase 1 : 56.00 at 20.0°C amp 042100 100% a.s. phase 2 : 26.10 at 25.3°C amp 023300 Date (ddmmyy) : 300103 Pressure (mBar) : 1013 FIRMWARE Code ver 1.081 : 01/23/03, 17:59:49 Xilinx built : 20/08/02 (MM/DD/YY) Reset condition : SLEEP *** *** *** ***

*******MEASURE MODE SETTINGS** Dynamic Averaging 1 measure mode 1 sec

start time	19:33:54					
date	time/hh:mm:ss	logtime/min	oxygen/% airsatur.	phase/°	amp	temp/°C
11.02.03	19:47:33	0	103.43	26.32	14894	22.5
11.02.03	19:47:33	0.015	104.18	26.24	14914	22.5
11.02.03	19:47:35	0.032	103.51	26.31	14938	22.5
11.02.03	19:47:36	0.05	103.02	26.36	14930	22.5
11.02.03	19:47:37	0.066	102.4	26.42	14939	22.5
11.02.03	19:47:38	0.083	101.23	26.54	14941	22.5
11.02.03	19:47:39	0.101	105.03	26.16	14890	22.5
11.02.03	19:47:40	0.117	103.96	26.27	14910	22.5
11.02.03	19:47:41	0.135	105.58	26.11	14955	22.5
11.02.03	19:47:42	0.152	103.65	26.3	14872	22.5
11.02.03	19:47:43	0.168	104.02	26.26	14896	22.5
11.02.03	19:47:44	0.182	105.02	26.16	14881	22.5
11.02.03	19:47:45	0.199	104.41	26.22	14864	22.5
11.02.03	19:47:46	0.217	103.99	26.26	14888	22.5
11.02.03	19:47:47	0.233	102.36	26.42	14879	22.5
11.02.03	19:47:48	0.251	103.91	26.27	14920	22.5
11.02.03	19:47:49	0.268	104.54	26.21	14886	22.5
11.02.03	19:47:50	0.285	103.66	26.29	14898	22.5
11.02.03	19:47:51	0.302	103	26.36	14870	22.5
11.02.03	19:47:52	0.319	104.76	26.19	14880	22.5
11.02.03	19:47:53	0.336	104.31	26.23	14886	22.5
11.02.03	19:47:54	0.353	104.05	26.26	14872	22.5
7 Calibration of Oxygen-Sensitive Fiber-Optic Minisensors

This chapter describes the calibration of oxygen minisensors containing a oxygen-sensitive coating (measuring range 0 - 500 % air-saturation). To calibrate sensors containing a coating you have to use the software oxymini01.exe. For any question, please contact our service team.

7.1 Calibration of a 2 mm POF Coated with a Oxygen-Sensitive Foil

7.1.1 Preparation of the Calibration Standards

Calibration of oxygen minisensors is performed using a conventional two-point calibration in **oxygen-free water** (cal 0) and **water-vapor saturated air** or **air-saturated water** (cal 100).

Preparation of calibration solution 0 (oxygen-free water):

- 1. Add one gram sodium sulfite (Na₂SO₃) to the vessel and label it *cal* 0;
- 2. Dissolve Na₂SO₃ in 100 mL water.

Water becomes oxygen-free due to a chemical reaction of oxygen with Na_2SO_3 . Additional oxygen, diffusing from air into the water, is removed by surplus of Na_2SO_3 .

3. Close the vessel with a screw top and shake it for approximately one minute to dissolve Na_2SO_3 and to ensure that water is oxygen-free.

Close the vessel after calibration with a screw top to minimize oxygen contamination

To prepare oxygen-free water you also can use sodium dithionite ($Na_2S_2O_4$). The shelf life of *cal 0* is about 24 hours provided that the vessel has been closed with the screw top.

Preparation of calibration standard 100 (water-vapor saturated air)

- 1. Place wet cotton wool in the vessel with the label *cal 100*.
- 2. Drill two holes for inserting the minisensor and the temperature sensor in the screw top and close the vessel.
- 3. Wait about 2 minutes to ensure that air is water-vapor saturated

Preparation of calibration solution 100 (air-saturated water)

- 1. Add 100 mL water to a suitable vessel and label it *cal 100*.
- 2. To obtain air-saturated water, blow air into the water using an air-pump with a glass-frit (airstone), creating a multitude of small air bubbles, while stirring the solution.
- 3. After 20 minutes, switch of the air-pump and stir the solution for further 10 minutes to ensure that water is not supersaturated.

7.1.2 Mounting the Oxygen-Sensitive Minisensors

- 1. Remove the oxygen sensor carefully from the protective cover.
- 2. Carefully remove the protective plastic cap covering the oxygen-sensitive sensor spot.
- 3. Fix the oxygen sensor with a clip to a laboratory support or a similar stable construction.
- 4. Remove the protective cap from the male fiber plug and connect it to the SMA-plug of the OxyMini device. The safety nut must be carefully attached while turning slightly clockwise.

7.1.3 Calibration without Automatic Temperature Compensation

Using the software, you can choose whether to perform the measurement and calibration with or without temperature compensation. If you want to perform the calibration without automatic temperature compensation, please ensure that the delivered temperature sensor TEMP 100 is not connected to the OxyMini.

Please note:

Calibration without temperature compensation only makes sense if there is no temperature change during the calibration of the oxygen sensor. Besides, it must be ensured that the temperature during later measurement is constant and already known. However, the temperatures during the measurement and the calibration process are allowed to be different.

- 1. Connect the OxyMini via the RS232 cable to your computer.
- 2. Switch on the OxyMini and connect the oxygen minisensor as shown in Chapter 7.1.2 "*Mounting the Oxygen-Sensitive Minisensor*".
- 3. Start the OxyMini software on your computer and click the **calibration** menu item.
- 4. Select the calibration routine: '*calibrate without temp sensor*' and click the '*cal.*' button

Measurement Calibration	
calibrate with temperature sensor	cal.
calibrate without temp. sensor	cal.
calibrate manually	manual

5. Enter the actual atmospheric pressure and the temperature of the calibration standards. The atmospheric pressure of the calibration is needed to convert the oxygen unit % air-saturation into partial pressure units (hPa, Torr) or concentrations units (mg/L μmol/L). Please note, that changes in the actual atmospheric pressure have no effect on the partial pressure units (hPa, Torr) and concentrations units (mg/L, μmol/L) but the oxygen units % air-saturation and % oxygen-saturation have to be corrected for air pressure changes.

	Calibration Menu
2 Point calibration - without temperature sensor	
atm. pressure	1013 🔮 mbar
1st point : 0 %air.sat.	phase temperature 56_50 ° ▲22_0 ▲ °C ✓ Store current value
2nd point : 100 %air.sat.	phase temperature 27 .57 ° .20 .0 . C Store current value
amplitude 27852	phase 27.67°
	✓ <u>F</u> inish <u>X</u> <u>C</u> ancel

6. Place the calibration standard 100 (*cal 100*), containing wet cotton wool, underneath the oxygen minisensor. The vessel with the label "*cal 100*" has to be closed with the screw top containing the two holes.

Insert the plastic fiber carefully through one of the holes without touching the oxygensensitive spot until it is about 3 cm deep inside the vessel.

Make sure that the plastic fiber with its sensor spot cannot touch the vessel and the cotton wool.



7. Wait about 2 minutes until the phase angle is constant (the variation of the phase angle should be smaller than $\pm 0.05^{\circ}$) and press the '*Store current value*' button to store the **100% air-sat.** and **temp. at 100%** values.

A message window opens and informs you that you will overwrite the existing calibration values. Click the '**Continue**' button to store the new calibration data.

Calibration Menu	
2 Point calibration - without temperature sensor	
atm. pressure 1013 🚔 mbar	
1st point :phasetemperature0 %air.sat.5728220CStore current value	
2nd point : phase temperature 100 %air.sat. 27 .57 ° ★ 20 .0 ★ ℃ ✓ Store current value	
amplitude phase 24681 57.28°	
<u>✓ F</u> inish <u>X C</u> ancel	
Calibration Message	
This will overwrite the exsisting calibration values.	

8. Now you have to record the second calibration value, oxygen-free water (*cal 0*). Place the vessel with the label "*cal 0*" underneath the oxygen minisensor. Insert the plastic fiber about 2 cm deep into the *cal 0* solution. Make sure that the plastic fiber with its sensor spot cannot touch the vessel. To increase the response time, stir the *cal 0* solution. Wait about 3 minutes until the phase angle is constant (the variation of the phase angle should be smaller than ± 0.05°) and click the '*Store current value*' button to store the 0% air-sat. and temp. at 0% values.



- 9. Now, calibration is complete. Confirm the calibration values by clicking the '*Finish'* button.
- 10. Wash the plastic fiber with its sensor spot with distilled water to clean it from sodium sulfite. Exchange the calibration solution 0 with an identical vessel filled with distilled water. Make sure, not to touch the sensor spot. Dip the plastic fiber with its sensor spot about 2 cm into a stirred washing solution for about 1 minute. Afterwards, retract the plastic fiber from the vessel without touching the sensor spot.
- 11. Protect the sensor spot with the delivered protective plastic cover and don't remove it again until just before measurement.

7.1.4 Calibration with Automatic Temperature Compensation

- 1. Connect the OxyMini via the RS232 cable to your computer.
- 2. To perform temperature compensated measurement, connect the temperature sensor TEMP 100 to the 4-pin connector in front of the OxyMini. Fix the temperature sensor and make sure that neither the temperature sensor nor its cable can touch the minisensor.
- 3. Switch on the OxyMini and connect the sensor as shown in Chapter 7.1.2 "*Mounting the Oxygen Minisensor*".
- 4. Start the OxyMini software on your computer and click the **calibration** menu item.
- 5. Select the calibration routine: '*calibrate with temperature sensor*' by clicking the '*cal.*' button.

Measurement Calibration	
calibrate with temperature sensor	cal.
calibrate without temp. sensor	cal.
calibrate manually	manual

6. Enter the 'actual atmospheric pressure'. The atmospheric pressure of the calibration is needed to convert the oxygen unit % air-saturation in partial pressure units (hPa, Torr) or concentrations units (mg/L μmol/L). Please note, that changes in the actual atmospheric pressure have no effect on the partial pressure units (hPa, Torr) and concentrations units (mg/L, μmol/L) but the oxygen units % air-saturation and % oxygen-saturation have to be corrected for air pressure changes.

	Calibration Menu	
2 Point cali	2 Point calibration - with temperature sensor	
atm. pressure	1013 👤 mbar	
1st point : 0 %air.sat.	phase temperature 56 .84 ° 20 .5 ℃ Store current value	
2nd point : 100 %air.sat.	phase temperature 28 .02 ° 20 .6 ℃ ✓ Store current value	
amplitude 31077	phase temperature 27.91 ° 20.6 °C	
	✓ <u>F</u> inish X <u>C</u> ancel	

 Place the calibration standard 100 (*cal 100*), containing wet cotton wool, underneath the oxygen minisensor. The vessel with the label "*cal 100*" has to be closed with the screw top containing the two holes.

Insert the plastic fiber carefully through one of the holes without touching the oxygensensitive spot until it is about 3 cm deep inside the vessel.

Make sure that the plastic fiber with its sensor spot cannot touch the vessel and the cotton wool.

Ensure that the temperature sensor is also inserted about 1-2 cm into the calibration vessel.



8. Wait about 1 minute until the phase angle and the temperature value is constant (the variation of the phase angle and the temperature should be smaller than ± 0.05° and 0.1 °C, respectively) and press the 'Store current value' button to store both the '100% air-sat.' and the temperature 'temp at 100%'.

A message window opens and informs you that you will overwrite the existing calibration values. Click the '*Continue*' button to store the new calibration data.

Calibration Message	×
This will overwrite the exsisting calibration values.	
Continue X Cancel	

9. Now you have to record the second calibration value, oxygen-free water (*cal 0*). Place the vessel with the label "*cal 0*" underneath the oxygen minisensor.

Insert the plastic fiber about 2 cm deep into the *cal 0* solution. Insert the temperature sensor into the *cal 0* calibration vessel.

Make sure that the plastic fiber with its sensor spot cannot touch the vessel.

To increase the response time, stir the *cal 0* solution. Wait about 3 minutes until the phase angle and the temperature is constant (the variation of the phase angle and the temperature should be smaller than $\pm 0.05^{\circ}$ and 0.1 °C, respectively) and click the '*Store current value*' button to store the '**0% air-sat**.' and **temp. at 0%** values.



	Calibration Menu	
2 Point cali	2 Point calibration - with temperature sensor	
atm. pressure	1013 📮 mbar	
1st point : 0 %air.sat.	phase temperature 58_76 ° 20_0 ℃ ✓ Store current value	
2nd point : 100 %air.sat.	phase temperature 26 .10 ° 25 .3 ℃ ✓ Store current value	
amplitude 16914	phase temperature 26.36 ° 22.4 °C	
	✓ Einish X Cancel	

- 10. Now, calibration with temperature compensation is complete. Confirm the calibration values by clicking the '*Finish*' button.
- 11. Wash the plastic fiber with its sensor spot and the temperature sensor with distilled water to clean it from sodium sulfite. Exchange the calibration solution 0 with an identical vessel filled with distilled water. Make sure, not to touch the sensor spot. Dip the plastic fiber with its sensor spot and the temperature sensor about 2 cm into a stirred washing solution for about 1 minute. Afterwards, retract the plastic fiber from the vessel without touching the sensor spot.
- 12. Protect the sensor spot with the delivered protective plastic cover and don't remove it again until just before measurement.

7.1.5 Manual Calibration

A manual calibration should be applied, if you don't want to calibrate your sensor again. However, this is only possible if you already know the calibration values of the special sensor.

- 1. Connect the OxyMini via the RS232 cable to your computer.
- 2. Switch on the OxyMini oxygen meter
- 3. Start the OxyMini software on your computer and click the **Calibration** menu item.
- 4. Select the calibration routine 'calibrate manually' and click the manual button

Measurement Calibration	
calibrate with temperature sensor	cal.
calibrate without temp. sensor	cal.
calibrate manually	manual

5. Enter the **atmospheric pressure** at which calibration was performed (not the **actual** one) and the respective calibration values **0% air-sat., temp. at 0%** and **100 % air-sat., temp. at 100%**.

	Calibration Menu
2 Point o	alibration - user defined
atm. pressure	1013 👤 mbar
1st point : 0 %air.sat.	phase temperature ★56.84 ★° ★20.9 ★°C
2nd point : 100 %air.sat.	phase temperature ▲28.02 ★ ° ★ 20.6 ★ °C
	✓ Einish X Cancel

6. Now, user-defined calibration is complete. Confirm the calibration values by clicking the **Finish** button. A message window opens and informs you that you will overwrite the existing calibration values. Click the '*Continue*' button to store the new calibration data.

Calibration Message	×
This will overwrite the exsisting calibration values.	
Continue X Cancel	

7.2 Calibration of Flow-Through Cell with Integrated Oxygen Sensor

7.2.1 Preparation of the Calibration Standards

Calibration of the minisensors is performed using a conventional two-point calibration in *oxygen-free water* (cal 0) and *air-saturated water* (cal 100).

Preparation of calibration solution 0 (oxygen-free water):

- 1. Add one gram sodium sulfite(Na₂SO₃) to the vessel and label it *cal* 0;
- Dissolve Na₂SO₃ in 100 mL water.
 Water becomes oxygen-free due to a chemical reaction of oxygen with Na₂SO₃. Additional oxygen, diffusing from air into the water, is removed by surplus of Na₂SO₃.
- 3. Close the vessel with a screw top and shake it for approximately one minute to dissolve Na₂SO₃ and to ensure that water is oxygen-free.

Close the vessel after calibration with a screw top to minimize oxygen contamination

To prepare oxygen-free water you also can use sodium dithionit ($Na_2S_2O_4$). The shelf life of *cal 0* is about 24 hours provided that the vessel has been closed with the screw top.

Preparation of calibration solution 100 (air-saturated water)

- 1. Add 100 mL water to a suitable vessel and label it *cal 100*.
- 2. To obtain air-saturated water, blow air into the water using an air-pump with a glass-frit (airstone), creating a multitude of small air bubbles, while stirring the solution.
- 3. After 20 minutes, switch of the air-pump and stir the solution for further 10 minutes to ensure that water is not supersaturated.

7.2.2 Mounting the Flow-Through Cell Oxygen Sensors

 Remove the flow-through cell oxygen sensor carefully from the protective cover. The oxygen-sensitive material is immobilized to a glass tube which is located in a Tconnector with two female Luer-Lock adapters. The glass tube is tightened with a viton seal and two male Luer-Lock connectors fix the glass tube in the T-connector. On request the seal can also be made from silicone.

Don't remove the two male Luer-Lock adapters from the T-connector. You may loose the viton seal and the glass tube may be dislocated.



2. Fix the male Luer-Lock adapter with the integrated 2 mm POF (polymer optical fiber) to the Luer T-connector and ensure that the fiber is located close to the glass tube.



- 3. Fix the flow-through cell with a clip to a laboratory support or a similar stable construction.
- 4. Connect the two male Luer-Lock connectors with the tubings of your flow-through system.
- 5. Remove the protective cap from the male fiber plug and connect it to the SMA-plug of the OxyMini device. The safety nut must be carefully attached while turning slightly clockwise.

7.2.3 Calibration without Automatic Temperature Compensation

Using the software, you can choose whether to perform the measurement and calibration with or without temperature compensation. If you want to perform the calibration without automatic temperature compensation, please ensure that the delivered temperature sensor TEMP 100 is not connected to the OxyMini oxygen meter.

Please note:

Calibration without temperature compensation only makes sense if there is no temperature change during the calibration of the oxygen sensor. Besides, it must be ensured that the temperature during later measurement is constant and already known. However, the temperatures during the measurement and the calibration process are allowed to be different.

- 1. Connect the OxyMini via the RS232 cable to your computer.
- 2. Switch on the OxyMini and connect the sensor as shown in Chapter 7.2.2 "*Mounting the Flow-Through Cell Oxygen Sensor*".
- 3. Start the OxyMini software on your computer and click the **calibration** menu item.
- 4. Select the calibration routine: 'calibrate without temp. sensor' and click the 'cal.' button

Measurement Calibration	
calibrate with temperature sensor	cal.
calibrate without temp. sensor	cal.
calibrate manually	manual

5. Enter the 'actual atmospheric pressure' and the 'temperature' of the calibration standards. The atmospheric pressure of the calibration is needed to convert the oxygen unit % air-saturation into partial pressure units (hPa, Torr) or concentrations units (mg/L μmol/L). Please note, that changes in the actual atmospheric pressure have no effect on the partial pressure units (hPa, Torr) and concentrations units (mg/L, μmol/L) but the oxygen units % air-saturation and % oxygen-saturation have to be corrected for air pressure changes.

	Calibration Menu	
2 Point cali	2 Point calibration - without temperature sensor	
atm. pressure	1013 📑 mbar	
1st point : 0 %air.sat.	phase temperature 56_50 ° ▲22_0 ▲ ℃ ✓ Store current value	
2nd point : 100 %air.sat.	phase temperature 27_57 ° ↓20_0 ↓ ℃ ✓ Store current value	
amplitude 27852	phase 27.67 °	
	✓ <u>F</u> inish X <u>C</u> ancel	

6. Connect one plastic tubing with a syringe, the other dip into the vessel containing the calibration solution 100, *cal 100*. Fill the syringe slowly with calibration solution 100. Please ensure that there are no air-bubbles located in the glass tube of the flow-through cell.





7. Wait about 1 minute until the phase angle is constant (the variation of the phase angle should be smaller than $\pm 0.05^{\circ}$) and press the '**Store current value**' button to store the **100% air-sat.** and **temp. at 100%** values. Afterwards, press the calibration solution into the waste.

A message window opens and informs you that you will overwrite the existing calibration values. Click the '*Continue*' button to store the new calibration data.

8. To record the second calibration value, oxygen-free water, dip the plastic tubing into the vessel containing the calibration solution 0, *cal 0* and fill the syringe slowly with it. Please ensure that there are no air-bubbles located in the glass tube of the flow-through cell.

Wait about 2 minutes until the phase angle is constant (the variation of the phase angle should be smaller than \pm 0.05°) and click the '**Store** *current value*' button to store the **0% air-sat.** and **temp. at 0%** values. Afterward, press the calibration solution into the waste.

	Calibration Menu
2 Point calibration - without temperature sensor	
atm. pressure	1013 🔮 mbar
1st point : 0 %air.sat.	phase temperature 57 .28 °
2nd point : 100 %air.sat.	phase temperature 27 .57 ° .20 .0 . Store current value
amplitude 24681	phase 57.28 °
	✓ <u>F</u> inish X <u>C</u> ancel

- 9. Now, calibration is complete. Confirm the calibration values by clicking the '*Finish'* button.
- 10. Now you have to wash the flow-through cell with distilled water to clean it from sodium sulfite. Dip the plastic tubing into a vessel containing distilled water and fill the syringe. Press the washing solution to the waste. Repeat this washing procedure 3 times.

7.2.4 Calibration with Automatic Temperature Compensation

- 1. Connect the OxyMini via the RS232 cable to your computer.
- 2. To perform temperature compensated measurement, connect the temperature sensor TEMP 100 to the 4-pin connector in front of the OxyMini.
- 3. Switch on the OxyMini and connect the sensor as shown in Chapter 7.2.2 "*Mounting the Flow-Through Cell Oxygen Sensor*".
- 4. Start the OxyMini software on your computer and click the calibration menu item.
- 5. Select the calibration routine: '*calibrate with temperature sensor*' and click the '*cal.*' button.

Measurement Calibration	
calibrate with temperature sensor cal .	
calibrate without temp. sensor	cal.
calibrate manually	manual

- 6. Enter the 'actual atmospheric pressure'. The atmospheric pressure of the calibration is needed to convert the oxygen unit % air-saturation into partial pressure units (hPa, Torr) or concentrations units (mg/L μmol/L). Please note, that changes in the actual atmospheric pressure have no effect on the partial pressure units (hPa, Torr) and concentrations units (mg/L, μmol/L) but the oxygen units % air-saturation and % oxygen-saturation have to be corrected for air pressure changes.
- Connect one plastic tubing with a syringe, the other dip into the vessel containing the calibration solution 100, "*cal 100*". Fill the syringe slowly with calibration solution 100. Please ensure that there are no air-bubbles located in the glass tube of the flow-through cell.

Insert the temperature sensor into the calibration solution "*cal 100*". Ensure that there is no temperature gradient between the flow-through cell and the temperature sensor.





8. Wait about 1 minute until the phase angle and the temperature value is constant (the variation of the phase angle and the temperature should be smaller than ± 0.05° and 0.2 °C, respectively) and press the 'Store current value' button to store both the 100% air-sat. and its temperature 'temp at 100%'. Afterwards, press the calibration solution back the waste.

A message window opens and informs you that you will overwrite the existing calibration values. Click the '*Continue*' button to store the new calibration data.

Calibration Menu	
2 Point calibration - with temperature sensor	
atm. pressure	1013 🚔 mbar
1st point : 0 %air.sat.	phase temperature 58 .76 ° 20 .0 ℃ Store current value
2nd point : 100 %air.sat.	phase temperature 26 .10 ° 25 .3 ℃ ✓ Store current value
amplitude 16435	phase temperature 26.05 ° 22.6 °C
	✓ <u>F</u> inish X <u>C</u> ancel

9. To record the second calibration value, oxygen-free water, dip the plastic tubing into the vessel containing the calibration solution 0, *cal 0* and fill the syringe slowly with it. Please ensure that there are no air-bubbles located in the glass tube of the flow-through cell.

Insert the temperature sensor into the calibration solution *cal* **0**. Ensure that there is no temperature gradient between the flow-through cell and the temperature sensor.



10. Wait about 2 minutes until the phase angle and the temperature is constant (the variation of the phase angle and temperature should be smaller than ± 0.05° and 0.2 °C, respectively) and click the 'Store current value' button to store the 0% air-sat. and temp. at 0% values. Afterwards, press the calibration solution into the waste. Again, a message window opens and informs you that you will overwrite the existing calibration values. Click the 'Continue' button to store the new calibration data.

Calibration Menu		
2 Point cali	2 Point calibration - with temperature sensor	
atm. pressure	1013 🚔 mbar	
1st point : 0 %air.sat.	phase temperature 56 .84 ° 20 .5 ℃ ✓ Store current value	
2nd point : 100 %air.sat.	phase temperature 28_02 ° 20_6 ℃ ✓ Store current value	
amplitude 31077	phase temperature 27.91 ° 20.6 °C	
	✓ <u>F</u> inish X <u>C</u> ancel	



- 11. Now, calibration with temperature compensation is complete. Confirm the calibration values by clicking the '*Finish'* button.
- 12. Now you have to wash the flow-through cell with distilled water to clean it from sodium sulfite. Dip the plastic tubing into a vessel containing distilled water and fill the syringe. Press the washing solution to the waste, not back into the vessel. Please repeat this washing procedure 3 times.

Also wash the temperature sensor by dipping it into water.

7.2.5 Manual Calibration

A manual calibration should be applied, if you don't want to calibrate your sensor again. However, this is only possible if you already know the calibration values of the special sensor.

- 1. Connect the OxyMini via the RS232 cable to your computer.
- 2. Switch on the OxyMini oxygen meter
- 3. Start the OxyMini software on your computer and click the **Calibration** menu item.
- 4. Select the calibration routine 'calibrate manually' and click the manual button

Measurement Calibration	
calibrate with temperature sensor	cal.
calibrate without temp. sensor	cal.
calibrate manually	manual

5. Enter the **atmospheric pressure** at which calibration was performed (not the **actual** one) and the respective calibration values **0% air-sat., temp. at 0%** and **100 % air-sat., temp. at 100%**.

	Calibration Menu
2 Point o	alibration - user defined
atm. pressure	1013 🚔 mbar
1st point :	phase temperature
0 %air.sat.	<u> </u>
2nd point : 100 %air.sat.	phase temperature → 28 - 02 ★ ° ★ 20 - 6 ★ °C
	<u>Finish</u>

6. Now, user-defined calibration is complete. Confirm the calibration values by clicking the **Finish** button. A message window opens and informs you that you will overwrite the existing calibration values. Click the '**Continue**' button to store the new calibration data.

Calibration Message	×
This will overwrite the exsisting calibration values.	
Continue X Cancel	

7.3 Calibration Planar Oxygen-Sensitive Foils Integrated in Glass Vials

7.3.1 Preparation of the Calibration Standards

Calibration of the minisensors is performed using conventional two-point calibration in *oxygen-free water* (cal 0) and *water-vapor saturated air* or *air-saturated water* (cal 100).

Preparation of calibration solution 0 (oxygen-free water):

- 1. Add one gram sodium sulfite (Na_2SO_3) to the vessel and label it cal 0;
- 2. Dissolve Na₂SO₃ in 100 mL water.

Water becomes oxygen-free due to a chemical reaction of oxygen with Na₂SO₃. Additional oxygen, diffusing from air into the water, is eliminated (removed) by surplus of Na₂SO₃.

3. Close the vessel with a screw top and shake it for approximately one minute to dissolve Na₂SO₃ and to ensure that water is oxygen-free.

Close the vessel after calibration with a screw top to minimize oxygen contamination To prepare oxygen-free water you also can use sodium dithionite ($Na_2S_2O_4$). The shelf life of **cal 0** is about 24 hours provided that the vessel has been closed with the screw top.

Preparation of calibration standard 100 (water-vapor saturated air)

- 1. Place wet cotton wool in the vessel and label it **cal 100**.
- 2. Drill two holes for inserting the minisensor and the temperature sensor in the screw top and close the vessel with it.
- 3. Wait about 2 minutes to ensure that air is water-vapor saturated

Preparation of calibration solution 100 (air-saturated water)

- 1. Add 100 mL water to a suitable vessel and label it *cal 100*.
- 2. To obtain air-saturated water, blow air into the water using an air-pump with a glass-frit, creating a multitude of small air bubbles, while stirring the solution.
- 3. After 20 minutes, switch of the air-pump and stir the solution for further 10 minutes to ensure that water is not supersaturated.

7.3.2 Mounting Planar Oxygen-Sensitive Foils

- 1. Remove the oxygen-sensitive foil carefully from the protective cover.
- 2. Glue small spots of the oxygen-sensitive foil into the desired glass vessel.





3. Remove the protective cap from the male fiber plugs of the delivered fiber cable and connect it to the SMA-plugs of the OxyMini device and the holding device. The safety nut must be carefully attached while turning slightly clockwise.



7.3.3 Calibration without Automatic Temperature Compensation

Using the software, you can choose whether to perform the measurement and calibration with or without temperature compensation. If you want to perform the calibration without automatic temperature compensation, please ensure that the delivered temperature sensor TEMP 100 is not connected to the OxyMini.

Please note:

Calibration without temperature compensation only makes sense if there is no temperature change during the calibration of the oxygen minisensor. Besides, it must be ensured that the temperature during later measurement is constant and already known. However, the temperatures during the measurement and the calibration process are allowed to be different.

- 1. Connect the OxyMini via the RS232 cable to your computer.
- 2. Switch on the OxyMini and connect the oxygen minisensor as shown in Chapter 7.3.2 "*Mounting Planar Oxygen-Sensitive Foils*".
- 3. Start the OxyMini software on your computer and click the **calibration** menu item.
- 4. Select the calibration routine: '*calibrate without temp. sensor*' and click the '*cal.*' button

Measurement Calibration	
calibrate with temperature sensor cal.	
calibrate without temp. sensor	cal.
calibrate manually	manual

5. Enter the actual 'atmospheric pressure' and the 'temperature' of the calibration standards. The atmospheric pressure of the calibration is needed to convert the oxygen unit % air-saturation into partial pressure units (hPa, Torr) or concentrations units (mg/L μmol/L). Please note, that changes in the actual atmospheric pressure have no effect on the partial pressure units (hPa, Torr) and concentrations units (mg/L, μmol/L) but the oxygen units % air-saturation and % oxygen-saturation have to be corrected for air pressure changes.

	Calibration Menu	
2 Point cali	2 Point calibration - without temperature sensor	
atm. pressure	1013 🚔 mbar	
1st point : 0 %air.sat.	phase temperature 56_50 ° ▲22_0 ▲ °C Store current value	
2nd point : 100 %air.sat.	phase temperature 27 .57 ° .20 .0 . Store current value	
amplitude 27852	phase 27.67°	
	✓ <u>F</u> inish X <u>C</u> ancel	

- 6. Place the calibration solution 100 (**cal 100**), air-saturated water (or water-vapor saturated air), into the glass vessel. To minimize the response time, slightly stir the solution. Please ensure that the **cal 100** solution completely covers the sensor foil.
- Wait about 30 sec. until the phase angle is constant (the variation of the phase angle should be smaller than ± 0.05°) and click the '*Store current value*' button to store the 100% air-sat. and temp. at 100% values.

8. Now you have to record the second calibration value, oxygen-free water (**cal 0**). Remove the *cal 100* solution from the vessel and fill it with the calibration standard 0, *cal 0*.

To minimize the response time, slightly stir the solution. Please note, vigorous stirring can lead to a oxygen contamination of the **cal 0** solution.

Wait about 3 minutes until the phase angle is constant (the variation of the phase angle should be smaller than \pm 0.05°) and press the '**Store current value**' button to store the **0% air-sat.** and **temp. at 0%** values.

Calibration Menu	
2 Point calibration - without temperature sensor	
atm. pressure	1013 🚔 mbar
1st point : 0 %air.sat.	phase temperature 57 . 28 ° . 22 . 0 . ℃ Store current value
2nd point : 100 %air.sat.	phase temperature 27 .57 ° .20 .0 . C ✓ Store current value
amplitude <mark>24681</mark>	phase <mark>57.28</mark> °
	✓ <u>F</u> inish X <u>C</u> ancel
Calibration Messa	ige X
This wil	Il overwrite the exsisting calibration values.

- 12. Now, calibration is complete. Confirm the calibration values by clicking the '*Finish*' button.
- 13. Wash the planar sensor foil with distilled water to clean it from sodium sulfite. Remove the calibration solution 0 and fill the vial with distilled water. Stir the solution for about 1 minute. Repeat this washing procedure for three times.

7.3.4 Calibration with Automatic Temperature Compensation

- 1. Connect the OxyMini via the RS232 cable to your computer.
- 2. To perform temperature compensated measurement, connect the temperature sensor TEMP 100 to the 4-pin connector in front of the OxyMini. Fix the temperature sensor and make sure that neither the temperature sensor nor its cable can touch the oxygen minisensor.
- 3. Switch on the OxyMini and connect the sensor as shown in Chapter 7.3.2 "*Mounting Planar Oxygen-Sensitive Foils*".
- 4. Start the OxyMini software on your computer and click the **calibration** menu item.
- 5. Select the calibration routine: '*calibrate with temperature sensor*' and click the '*cal.*' button.

Measurement Calibration	
calibrate with temperature sensor	cal.
calibrate without temp. sensor	cal.
calibrate manually	manual

- 6. Enter the 'actual atmospheric pressure'. The atmospheric pressure of the calibration is needed to convert the oxygen unit % air-saturation into partial pressure units (hPa, Torr) or concentrations units (mg/L μmol/L). Please note, that changes in the actual atmospheric pressure have no effect on the partial pressure units (hPa, Torr) and concentrations units (mg/L, μmol/L) but the oxygen units % air-saturation and % oxygen-saturation have to be corrected for air pressure changes.
- 7. Place the calibration solution 100 (**cal 100**), air-saturated water, into the glass vessel. To minimize the response time, slightly stir the solution.

Ensure that the temperature sensor has been dipped about 1-2 cm into the glass vessel containing the **cal 100**.

8. Wait about 3 minute until the phase angle and the temperature value is constant (the variation of the phase angle and the temperature should be smaller than ± 0.05° and 0.1 °C, respectively) and press the 'Store current value' button to store both the 100% air-sat. and its temperature temp at 100%.

A message window opens and informs you that you will overwrite the existing calibration values. Click the '*Continue*' button to store the new calibration data.

9. Now you have to record the second calibration value, oxygen-free water (**cal 0**). Remove the *cal 100* solution from the vessel and fill it with the calibration standard 0, *cal 0*.

Insert the temperature sensor into the glass vessel containing cal 0.

To minimize the response time, slightly stir the solution. Please note, vigorous stirring can lead to a oxygen contamination of the **cal 0** solution.

Wait about 3 minutes until the phase angle and the temperature is constant (the variation of the phase angle and the temperature should be smaller than \pm 0.05° and 0.1 °C, respectively) and click the '**Store** *current value*' button to store the **0% air-sat.** and **temp. at 0%** values.

	Calibration Menu
2 Point cali	bration - with temperature sensor
atm. pressure	1013 🛓 mbar
1st point : 0 %air.sat.	phase temperature 56 .84 ° 20 .5 ℃ ✓ Store current value
2nd point : 100 %air.sat.	phase temperature 28 .02 ° 20 .6 ℃ Store current value
amplitude 31077	phase temperature 27.91 ° 20.6 °C
	✓ <u>F</u> inish X <u>C</u> ancel
Calibration Message	
This	will overwrite the exsisting calibration values.

- 10. Now, calibration with temperature compensation is complete. Confirm the calibration values by clicking the '*Store*' button.
- 11. Wash the planar sensor foil with distilled water to clean it from sodium sulfite. Remove the **cal 0** and fill the vial with distilled water. Stir the solution for about 1 minute. Repeat this washing procedure for three times.

Wash also the temperature sensor with distilled water

7.3.5 Manual Calibration

A manual calibration should be applied, if you don't want to calibrate your sensor again. However, this is only possible if you already know the calibration values of the special sensor.

- 1. Connect the OxyMini via the RS232 cable to your computer.
- 2. Switch on the OxyMini oxygen meter
- 3. Start the OxyMini software on your computer and click the **Calibration** menu item.
- 4. Select the calibration routine 'calibrate manually' and click the manual button

Measurement Calibration	
calibrate with temperature sensor	cal.
calibrate without temp. sensor	cal.
calibrate manually	manual

5. Enter the **atmospheric pressure** at which calibration was performed (not the **actual** one) and the respective calibration values **0% air-sat., temp. at 0%** and **100 % air-sat., temp. at 100%**.

	Calibration Menu		
2 Point o	2 Point calibration - user defined		
atm. pressure	1013 糞 mbar		
1st point : O %air.sat.	phase temperature ♣56.84 ★° ★20.9 ★°C		
2nd point : 100 %air.sat.	phase temperature ▲ 28 .02 ★ ° ★ 20 .6 ★ °C		
	✓ <u>F</u> inish X <u>C</u> ancel		

 Now, user-defined calibration is complete. Confirm the calibration values by clicking the Finish button. A message window opens and informs you that you will overwrite the existing calibration values. Click the '*Continue*' button to store the new calibration data.



8 Measurement

Calibration of the sensor is recommended before each measurement (see chapter 7&8 "Calibration of Oxygen-Sensitive Fiber-Optic Minisensors"). If you don't want to recalibrate a sensor, you can use the calibration values of your last measurement (see "User-Defined Calibration").

Each calibration is only valid for the corresponding sensor and should be repeated at least after every new start of the OxyMini. Especially, after longer measurements (more than 10000 measure points or 3 h continuous mode) the sensor should be re-calibrated.

Ensure that the temperature of the sample is known and is constant during measurement, if you do not use temperature compensation. In the case of temperature-compensated

measurements, the temperature sensor TEMP 100 should be positioned as close as possible to the oxygen minisensor to avoid temperature differences.

8.1 Measurement with POFs Coated with an Oxygen-Sensitive Foil

- 1. Please carefully read instructions "*Calibration of a 2 mm POF Coated with an Oxygen-Sensitive Foil*" in the manual chapters 7.1. There you will find relevant information about the proper handling of the oxygen-sensitive sensors.
- 2. Connect the OxyMini via the RS232 cable to your computer and switch on both.
- 3. Connect the temperature sensor TEMP 100 to the 4-pin connector on the front panel of the OxyMini and carefully tighten the safety nut, to perform temperature-compensated measurement. Fix the temperature sensor.
- 4. Calibrate the sensor according to chapter 7.1 or 8.1 "*Calibration of a 2 mm POF Coated with an Oxygen-Sensitive Foil*". If you do not want to re-calibrate the sensor but use the calibration values of your last measurement, choose "*User-Defined*".
- 5. Position the oxygen-sensor in the sample. Ensure, that no air bubbles are located at the sensor and that the temperature sensor is located close to the sensor in the case of temperature compensated measurements.

8.2 Measurement with a Flow-Through Cell with Integrated Oxygen Sensor

- 1. Please carefully read chapter 7.2, 8.2 " *Calibration of a Flow-Through Cell with Integrated Oxygen Sensor* " *in* the manual instructions. There you will find relevant information about the proper handling of oxygen flow-through sensors. They are the basic for the following chapter.
- 2. Connect the OxyMini via the RS232 cable to your computer.
- 3. Fix the male Luer-Lock adapter with the integrated 2 mm POF (polymer optical fiber) to the Luer T-connector and ensure that the fiber is located close to the glass tube.



4. Connect the temperature sensor TEMP 100 to the 4-pin connector on the front panel of the OxyMini and carefully tighten the safety nut, to perform temperaturecompensated measurement. Immerse the temperature sensor in your sample and fix it with a laboratory support.

- Calibrate the sensor according to chapter 7.2 or 8.2 " Calibration of a Flow-Through Cell with Integrated Oxygen Sensor ". If you do not want to re-calibrate the sensor but use the calibration values of your last measurement, choose "User-Defined".
- 6. Connect the Luer-Lock adapter at the end-pieces of the T-connector with tubings and pump your sample through the flow-through cell.

Ensure, that no air bubbles are located in the flow through cell.

8.3 Measurement with Oxygen-Sensitive Foils

Integrated sensors are offered as single sensor spots of 25 mm² on a polyester or glass support or are already glued into sample flasks (e.g. cell culture flasks) containing a SMA holding device. Please contact our service team to find the optimal solution for your application.



Cell culture flasks containing a holding device to connect SMA fiber bundles to read out the oxygen concentration through the flask wall.

- 1. Please carefully read chapter 7.3 or 8.3 "*Calibration of Planar Oxygen-Sensitive Foils Integrated in Glass Vials*" in the manual instructions. There you will find relevant information about the proper handling of planar oxygen-sensitive foils integrated in glass vials. They are the basic for the following chapter.
- 2. Connect the OxyMini via the RS232 cable to your computer.

3. Remove the protective cap from the male fiber plugs of the delivered fiber cable and connect it to the SMA-plugs of the OxyMini device and the holding device. The safety nut must be carefully attached while turning slightly clockwise.



- 4. Connect the temperature sensor TEMP 100 to the 4-pin connector on the front panel of the OxyMini and carefully tighten the safety nut, to perform temperature-compensated measurement. Immerse the temperature sensor into the glass vial with your sample and fix it with a laboratory support.
- 5. Calibrate the sensor according to chapter 7.3 or 8.3 " Calibration of Planar Oxygen-Sensitive Foils Integrated in Glass Vials". If you have obtained sterile glass vials with an integrated oxygen sensor and hence are not able to calibrate the sensor, use the precalibration values of the inspection sheet you have obtained with the planar oxygensensitive foil. Choose "User-Defined" calibration.

8.4 Some Advice for Correct Measurement

8.4.1 Signal drifts due to oxygen gradients

Please, keep in mind, that the sensor only measures the oxygen content near its surface. In unstirred solutions occurs often an oxygen concentration gradient.

Please check if air bubbles are on the sensor tip whenever unexpected drifts, gradients or unstable measurement values occur. Critical conditions for bubble formations are, for example, purging with air or other gases and increasing temperature during measurement.

The formation of a bio-film during long term measurements or the accumulation of other sample components like oil or solid substances may lead to an oxygen gradient.

8.4.2 Signal drifts due to temperature gradients

A further source of imprecise measurement is insufficient temperature compensation. If you use the temperature compensation, ensure that no temperature gradients exist between the oxygen sensor and the temperature sensors. If you measure without temperature compensation, please keep in mind that the OxyMini only measures correctly, if the sample temperature is constant during measurement and the temperature is the same as you typed in at the beginning of the measurement. Please also refer to Chapter 14.5 "Formulas for temperature compensation". If the temperature is measured with a precision of \pm 0.2 °C, there is a variation in the measuring value at 100% air-saturation of \pm 0.7 % air-saturation. Please choose the measurement with temperature compensation to minimize temperature gradients.

8.4.3 Signal drift due to photo-decomposition

The oxygen-sensitive material may be subject to photo-decomposition resulting in a signal drift. Photo-decomposition takes place only during illumination of the sensor tip and depends on the intensity of the excitation light. Therefore, the excitation light was minimized.

Continuous illumination of a **POF-** oxygen sensor over a period of 24 hours may lead to a phase drift of up to + 0.4 % air-saturation measured at 100% air-saturation at 20°C. However, this effect of photo-decomposition can even be minimized, by changing the measuring mode to the second or minute interval mode. In these modes, the software switches off the excitation light after recording the data point and switches it on after the interval you have chosen.

Please use the interval method whenever it is possible to increase the shelf life of the microsensor.

Drift in % air-saturation at 100% air-saturation when illuminating the oxygen sensor for 1, 12 and 24 hours in the continuous mode.

	Drift per hour	Drift per 12 hours	Drift per 24 hours
Oxygen minisensor			< 0.4 % air-saturation

Photo-decomposition test of oxygen sensitive, continuously illuminating the sensor membrane for 25 hours

8.4.4 Signal drift due to too much ambient light



A source of error is the detector overload due to too much ambient light. A detector overload can be recognized with the red shining warning light **overload**, which you can find at the right bottom of the window.

red: background light (e.g. direct sunlight, lamp) is too high. Decrease of false light by decreasing the light intensity or darkening the sample is necessary.

green: ratio of sensor signal to false light is acceptable

Please notify that your measurement is not reliable if the warning light overload is shining red. A detector overload causes a decrease in both amplitude and phase angle.

8.4.5 Performance proof

If you want to prove the performance during the past measurement, please check the calibration values by inserting the sensor tip in the 'cal 0' and 'cal 100' calibration standards when you have finished your measurement. If the device shows 0% air-saturation immersing the sensor tip into the 'cal 0' solution and 100 % air-saturation measuring the 'cal 100' standard, the sensor worked perfectly during the whole measurement.

8.4.6 Correction for air-pressure variations

The atmospheric pressure of the calibration is needed to convert the oxygen unit % airsaturation in partial pressure units (hPa, Torr) or concentrations units (mg/L µmol/L). The partial pressure and the oxygen concentrations units are calculated from % air-saturation by the software. Consequently, changes in the actual atmospheric pressure have no effect on the partial pressure units (hPa, Torr) and concentrations units (mg/L, µmol/L) but the oxygen units % air-saturation and % oxygen-saturation have to be corrected for air pressure changes.

9 General Instructions:

9.1 Warm-Up Time

The warm up time of the electronic and opto-electronic components of the OxyMini is 5 min. Afterwards stable measuring values are obtained.

9.2 Maintenance

The instrument is maintenance-free.

The housing should be cleaned only with a moist cloth. Avoid any moisture entering the housing! Never use benzine, acetone, alcohol or other organic solvents.

The SMA-fiber connector of the minisensor can be cleaned only with lint-free cloth. The Sensor tip may be rinsed only with distilled water. Please ensure, that no sample residues are inside the syringe needle. If necessary, rinse the glass-fiber and the syringe needle with distilled water.

9.3 Service

Balancing, maintenance and repair work may only be carried out by the manufacturer.

Please contact WPI's service team should you have any questions. We look forward for helping you.

10 Technical Data

10.1 General Data

MODES	
Oxygen minisensor	range: 0 - 500 % air-saturation
	resolution: 30 ± 0.3 % air -aturation
	100 \pm 0.9 % air-saturation
	250 \pm 3.6 % air-saturation
	accuracy: \pm 1 % air-saturation at 100 % air-saturation
temperature	range: 0 - 50 °C
	resolution: ± 0.5 °C
	accuracy: ± 1° C

CALIBRATION PROCEDURE	
Oxygen minisensor	2-point calibration in oxygen free water and humidified air or air-saturated water

OPTICAL OUTPUT / INPUT	
Optical connector	SMA compatible, 2 mm polymer optical fiber
Channels	1
Wavelength	505 nm

TEMPERATURE SENSOR INPUT

Lemo Connector Size 00	Connector for TEMP100 temperature sensor
	DC-Range : 12 V/1250mA up to 18V/900mA

DIGITAL OUTPUT		
communication protocoll	serial interface RS232 19200 Baud, Databits 8, Stoppbits 1, Parity none, Handshake none	
instrument output:	on RJ11 4/4 plug	
Interface cable to PC:	RJ11 4/4 to DSub9:	

ENVIRONMENTAL CONDITIONS	
Operating temperature	0 to +50°C
Storage temperature	-10 to +65°C
Relative humidity:	up to 95%

OPERATION CONTROL	LED at the front panel:	
	red: instrument off	
	green: instrument on	
	orange: stand by	
DIMENSIONS	length: 185 mm;	
	width: 110 mm;	
	height: 45 mm;	

10.2 Analog Output and External Trigger

The TX3-AOT –instrument version is supplied with a dual programmable 12bit analog output with galvanic isolation and an external trigger input.

weight: 630 g;

ANALOG OUTPUT

GENERAL SPECIFICATION - ANALOG OUTPUT

Channels Connector Resolution Output range Galvanic isolation Shortcut protection	2 BNC 12 bit 0 to 4095 mV 500 V rms Yes	(±2mV max. error)
Programmable to	oxygen, temperature, amplitude, phase by software	
Equivalence coefficients : oxygen temperature amplitude phase	1 :: 0.1 1 :: 0.1 1 :: 10 1 :: 0.025	(i.e.: 973 mV = 97.3 % air saturation) (i.e.: 208 mV = 20.8°C) (i.e.: 2220 mV = 22200 relative units) (i.e.: 1100 mV = 27.50°)

<u>Update rate:</u> The update rate is dependent on the sampling rate of the software. If an external trigger is used, the update rate is equivalent to the trigger pulse rate.

DC SPECIFICATION - ANALOG OUTPUT

Resolution

oxygen	$\pm 2mV \rightarrow \pm 0.2$ % air saturation
temperature	± 2mV ➔ ± 0.2°C
amplitude	$\pm 2mV \rightarrow \pm 20$ relative units
phase	± 2mV ➔ ± 0.05°)

± 10mV

Accuracy error

68
EXTERNAL TRIGGER INPUT

GENERAL SPECIFICATION - EXTERNAL TRIGGER INPUT

Channels	1
Connector	BNC
Input voltage range	TTL-compatible / up to 24V
Trigger mode	Low-High-Low
	(Input must be kept Low for at least 50µs)
Normal state	no current
Isolation	500V rms

Timing Specifications:

15 ns (see TTL-specification)
2 ms
3 ms
10 ms
13 ms

10.3 Technical Notes

Power Adapter

OxyMini should always be used with the original power adapter (110-220VAC/12VDC). As an alternative power source a battery can be used that meets the DC input voltage given in technical specification. The battery adapter cable is available as an additional accessory.

Analog Outputs

WARNING: The analog outputs are not protected against any input voltage! Any voltage applied to the analog outputs can cause irreversible damage of the circuit.

RS232 Interface

The unit uses special interface cable. Another cable can cause the unit's malfunction.

Optical Output (ST)

The ST connector is a high precision optical component. Please keep it clean and dry. Always use the rubber cap to close the output when not in use.

10.4 Operation Notes

Oxygen Measurement

To achieve the highest accuracy OxyMini should be warmed-up for 5min before starting the measurement. Please see the details of measurement process described in OxyMini manual.

Temperature Compensation

No other than supplied temperature sensor could be used with the unit. The use of any other temperature sensor can damage the oxygen meter.

11 Trouble Shooting

Error	Cause	Action
Device does not work and LED on the front panel is not lit	Device is not switched on;	Switch on device with ON/OFF switch on the rear panel;
	No power supply;	Connect power supply with device;
Device does not work and LED on the front panel is on	No connection to PC;	Check connection of the device to your PC (RS 232)
Temperature compensation failed, no temperature	TEMP 100 sensor is not connected;	Check connection; Contact our service:
measurement possible	TEMP 100 Sensor is faulty;	
Warning light:		
Amplitude: red	Oxygen sensor is not connected;	Check connection of the SMA- connector;
	Sensor spot is removed from the plastic fiber;	Replace oxygen sensor
Warning light:		
Phase: red	phase angle out of limits;	check connection of the oxygen sensor;
		replace oxygen sensor;
Warning light:		
overload: red	too much ambient light;	reduce ambient light by decreasing the light intensity;
		use optical-isolated oxygen sensors;
no cal	calibration failed;	calibrate again;
		check calibration solutions;
	Sensor was not in the right calibration standard;	Immerse sensor in the proper calibration vessel;
	sulfite solution has aged;	Prepare new sulfite solution;
Strong signal fluctuations	Air bubbles at sensor tip;	Remove air bubbles by carefully tapping;
	Too much ambient light;	reduce ambient light by decreasing the light intensity;

12 Appendix

12.1 Basics in Optical Sensing of Oxygen

12.1.1 Dynamic Quenching of Luminescence

The principle of measurement is based on the effect of dynamic luminescence quenching by molecular oxygen. The following scheme explains the principle of dynamic luminescence quenching by oxygen.



Figure 12.1 Principle of dynamic quenching of luminescence by molecular oxygen (1) Luminescence process in absence of oxygen (2) Deactivation of the luminescent indicator molecule by molecular oxygen

The collision between the luminophore in its excited state and the quencher (oxygen) results in radiationless deactivation and is called collisional or dynamic quenching. After collision, energy transfer takes place from the excited indicator molecule to oxygen which consequently is transferred from its ground state (triplet state) to its excited singlet state. As a result, the indicator molecule does not emit luminescence and the measurable luminescence signal decreases.

A relation exists between the oxygen concentration in the sample and the luminescence intensity as well as the luminescence lifetime which is described in the Stern-Volmer-equation (1). Here, τ_0 and τ are the luminescence decay times in absence and presence of oxygen (I₀ and I are the respective luminescence intensities), [O₂] the oxygen concentration and K_{SV} the overall quenching constant



(1)

- I: Luminescence intensity in presence of oxygen
- I₀: Luminescence intensity in absence of oxygen
- τ : Luminescence decay time in presence of oxygen
- τ_0 : Luminescence decay time in absence of oxygen
- K_{SV} : Stern-Volmer constant (quantifies the quenching efficiency and therefore the sensitivity of the sensor)
- [O₂]: oxygen content



Figure. 12.2 (A) Luminescence decrease in the presence of oxygen. (B) Stern-Volmer plot.

Indicator dyes quenched by oxygen are, for example polycyclic aromatic hydrocarbons, transition metal complexes of Ru(II), Os(II) and Rh(II), and phosphorescent porphyrins containing Pt(II) or Pd(II) as the central atom.

12.1.2 Major Components of Fiber-Optic Minisensors

In optical chemical sensors, the analyte interacts with an indicator and changes its optical properties. The result is either a change in the color (absorbance or spectral distribution) or the luminescence properties (intensity, lifetime, polarisation). Light acts as the carrier of the information.

The major components of a typical fiber optical sensing system are

- a light source to illuminate the sensor (laser, light emitting diode, lamps)
- an optical fiber as signal transducer (plastic or glass fiber)
- a photodetector (photodiode, photomultiplier tube, CCD-array)
- the optical sensor (indicator immobilised in a solid matrix)

Figure 12.3 Scheme of a minisensor.

Appendix

Figure 12.4 Schematic drawing of the optical setup of a measuring system with minisensors. (LED: light emitting diodes, PMT: photomultiplier, OF: optical filters, ST: fiber connector)

12.1.3 Advantages of Optical Oxygen-Sensitive Minisensors

- no oxygen is consumed during the measurement;
- · the signal is independent of changes in flow velocity;
- they are able to measure the oxygen content in dry gases
- they are insensible towards electrical interferences and magnetic fields;
- they are more sensitive than conventional electrodes (up to ppt-range);
- long-term stability and low drift;
- using silica fibers, it is possible to measure in samples while physically separate from the light source and detectors;
- light conducting fibers are able to transport more information than power currents (information can be simultaneously transferred, e.g., intensity of light, spectral distribution, polarisation, information such as decay time or delayed fluorescence);

12.1.4 Luminescence Decay Time

The OxyMini measures the luminescence decay time of the immobilised luminophore as the oxygen dependent parameter.

$$\tau = f([O_2]) \tag{2}$$

The OxyMini uses the phase-modulation technique to evaluate the luminescence decay time of the indicators. If the luminophore is exited with a sinusoidally intensity modulated light, its decay time causes a time delay in the emitted light signal. In technical terms, this delay is the phase angle between the exiting and emitted signal. This phase angle is shifted as a function of the oxygen concentration. The relation between decay time and the phase angle is shown by the following equation:

(3a)

$$\tan \Phi = 2\pi \cdot \mathbf{f}_{\mathrm{mod}} \cdot \tau \tag{3b}$$

$$\tau = \tan \Phi = \Phi = f([O_2])$$
(3c)

 τ : luminescence decay time; Φ : phase angle; f_{mod} : modulation frequency

73



Figure 12.5 Schematic of the single exponential decay ($t_0 > t_1$).

Figure 12.6 The luminophore is excited with sinusoidally modulated light. Emission is delayed in phase expressed by the phase angle F relative to the excitation signal, caused by the decay time of the excited state

The measurement of the luminescence decay time, an intrinsically referenced parameter, has the following advantages compared to the conventional intensity measurement.

- The decay time does not depend on fluctuations in the intensity of the light source and the sensitivity of the detector;
- The decay time is not influenced by signal loss caused by fiber bending or by intensity changes caused by changes in the geometry of the sensor;
- The decay time is, to a great extent, independent of the concentration of the indicator in the sensitive layer. Photobleaching and leaching of the indicator dye has no influence on the measuring signal;

The decay time is not influenced by variations in the optical properties of the sample including turbidity, refractive index and coloration.

12.2 Determination of the Oxygen Concentration Using a Modified Stern-Volmer Equation

The Stern-Volmer equation (4) displays a linear correlation between $tan\Phi_0/tan\Phi$ or τ_0/τ and the oxygen concentration [O₂].

(4)

- Φ_0 phase angle of oxygen-free water
- Φ measured phase angle
- K_{SV} Stern-Volmer Constant
- [O₂] oxygen content in %-air-saturation

The Stern-Volmer plots of all our sensors show a distinct non-linearity in their response behavior which is also observed for many other oxygen sensors described in literature. This non-linear response behavior can be described with a modified Stern-Volmer equation.

74

$$\frac{\tan \Phi_0}{\tan \Phi} = \left(\frac{f_1}{1 + K_{SV_1} \cdot [O_2]} + \frac{1 - f_1}{1 + K_{SV_2} \cdot [O_2]}\right)^{-1}$$
(5)

This model is based on the assumption that the indicator is distributed in the polymer matrix at two different sites and each fraction (f_1 , 1- f_1) shows a different quenching constant (K_{SV1} , K_{SV2}).

For practical use this model is not very convenient since it has too many parameters which have to be calibrated. Therefore, two simplified models based on equation 5 can be used.

In the first model, one fraction of the indicator is assumed to be non-quenchable (K_{SV2}=0).

$$\frac{\tan \Phi_0}{\tan \Phi} = \left(\frac{f_1}{1 + K_{SV} \cdot [O_2]} + (1 - f_1)\right)^{-1}$$
(6)

Equation 7 was obtained describing the oxygen calibration plot of a minisensor. The correlation coefficient R^2 of this fit was higher than 0.999.

The oxygen content in %-air-saturation can be calculated according to equation 8.

A second model, which also is based on equation 5, can be used for describing the oxygen calibration plot. In this model, K_{SV2} is set to be x^*K_{SV1} . f_1 was determined to be 0.808 and x was determined to be 1/29.87. The correlation coefficient R² of this fit was higher than 0.9999.

The oxygen content in %-air-saturation can be calculated according to equation 10.

$$[O_2] = \frac{-B + \sqrt{B^2 - 4 \cdot A \cdot C}}{2 \cdot A}$$
(10)

with the coefficients

$$\mathbf{A} = \frac{\tan \Phi}{\tan \Phi_0} \cdot \mathbf{x} \cdot \mathbf{K}_{\rm SV}^2 \tag{10a}$$

(9)

(7)

(8)

$$B = \frac{\tan \Phi}{\tan \Phi_0} \cdot K_{SV} + \frac{\tan \Phi}{\tan \Phi_0} \cdot x \cdot K_{SV} - f_1 \cdot x \cdot K_{SV} - K_{SV} + f_1 K_{SV}$$
(10b)
$$C = \frac{\tan \Phi}{\tan \Phi_0} - 1$$
(10c)

Please note:

These conversion formulas are only valid in aqueous solutions and humidified air. These formulas have to be modified if measurements have to be performed in organic solvents or solutions with high salinity.

% -Saturation

%-air saturation

Default-Setting of the instrument (see equation 10 in 14.2)

%-oxygen saturation

$$%O_2 = \% \text{ air - saturation} \cdot \frac{20.95}{100}$$
 (11)

0.2095: volume content of oxygen in air

ppm in the gaseous phase:

$$ppm[O_{2}] = \%air - saturation \cdot \frac{20.95}{100} \cdot 10000$$

$$lppm = \frac{1}{1000000} = \frac{lmg}{lkg} = \frac{l\mu L}{lL} = \frac{1}{10000} \%$$
(12)

Partial pressure of oxygen

$$p_{O_2}[hPa] = (p_{atm}[hPa] - p_W(T)[hPa]) \cdot \frac{\% \text{ air - saturation}}{100} \cdot 0.2095$$
(13)

in mbar

$$p_{O_2}[mbar] = (p_{atm}[mbar] - p_w(T)[mbar]) \cdot \frac{\% \text{ air - saturation}}{100} \cdot 0.2095$$
(14)

in Torr

Please note: 1 mbar = 1 hPa = 0.750 Torr (15)

Appendix

Oxygen Concentration

in mg/L

$$c_{O_{2}}[mg/L] = \frac{p_{atm} - p_{W}(T)}{p_{N}} \cdot \frac{\% \text{ air - saturation}}{100} \cdot 0.2095 \cdot \alpha(T) \cdot 1000 \cdot \frac{M(O_{2})}{V_{M}}$$
(16)

in ppm

$$c_{O_{2}}[ppm] = \frac{p_{atm} - p_{W}(T)}{p_{N}} \cdot \frac{\% \text{ air - saturation}}{100} \cdot 0.2095 \cdot \alpha(T) \cdot 1000 \cdot \frac{M(O_{2})}{V_{M}}$$
(17)

in μ mol/L

$$c_{O_2}[\mu mol/L] = \left[\frac{p_{atm} - p_W(T)}{p_N} \cdot \frac{\% \text{ air - saturation}}{100} \cdot 0.2095 \cdot \alpha(T) \cdot 1000 \cdot \frac{M(O_2)}{V_M}\right] \cdot 31.25$$
(18)

patm: actual atmospheric pressure;

p_N: standard pressure (1013 mbar);

0.2095: volume content of oxygen in air;

 $p_W(T)$: vapor pressure of water at temperature T given in Kelvin;

 α (T): Bunsen absorption coefficient at temperature T;

M(O₂): molecular mass of oxygen (32 g/mol);

V_M: molar volume (22.414 l/mol);

12.4 Temperature Dependent Constants Affecting the Oxygen Content

12.4.1 Water Vapor Pressure

As shown in equation 13 - 18, the water vapor pressure p_w influences the oxygen partial pressure of air-saturated water and water-vapor saturated air.

Oxygen partial pressure in dry air:

 $p(O_2)$: oxygen partial pressure in dry air at a barometric pressure p_{atm} 0.2095: volume content of oxygen in air;

Oxygen partial pressure in air-saturated water and water-vapor saturated air:

Water vapor pressure is strongly affected by temperature variations and this, of course, influence the oxygen partial pressure as shown in equation 19.

Table 12.1 Variation of water vapor pressure $p_W(T)$ with temperature.

θ [°C]	0	5	10	15	20	25	30	35	40	50
т [К]	273	278	283	288	293	298	303	308	131	323
p _w (T) [mbar]	6.1	8.7	12.3	17.1	23.3	31.7	42.4	56.3	73.7	123.3

A convenient fitting function is given by the Campbell equation 20

$$p_{W}(T) = \exp\left(A - \frac{B}{T} - C \cdot \ln T\right)$$

(21)

where T is the temperature in Kelvin and A, B and C constants given in Figure 14.7

Figure. 12.7 Variation of water vapor pressure with temperature.

(19)

(20)

12.4.2 Bunsen Absorption Coefficient

The solubility of oxygen in water is temperature dependent and can be described using the Bunsen absorption coefficient $\alpha(\theta)$ and the oxygen partial pressure $p(O_2)$ according to equation 22. With increasing temperature, the solubility of oxygen in water decreases.

$$c_{s}(p,\theta) = \frac{p(O_{2})}{p_{N}}\alpha(\theta)$$
(22)

 $c_{S}(p,\theta)$: temperature dependent solubility of oxygen in water

 $p(O_2)$: oxygen partial pressure

p_N: standard pressure (1013 mbar);

Table 12.2	Variation of Bunsen	absorption	coefficient $\alpha(0)$) with	temperature.
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θ [°C]	0	5	10	15	20	25	30	35	40	50
α(θ) [.] 10 ³	49.01	42.94	38.11	34.17	31.01	28.43	26.30	24.63	23.16	20.85

The data in Table 14.2 can be described by two forms of equations.

The first form of equation to describe the temperature dependent variation of the Bunsen absorption coefficient $\alpha(\theta)$ is obtained by fitting a general power series to the values in Table 14.2. A fourth degree polynomial fit can be chosen, yielding equation 23

$$10^{3} \alpha = a + b \cdot \theta + c \cdot \theta^{2} + d \cdot \theta^{3} + e \cdot \theta^{4}$$
(23)

where θ is the temperature in °C and a - e the coefficients calculated by standard curve fitting procedures.

The square of the correlation coefficient is 0.999996.

Figure 12.8 Variation of Bunsen absorption coefficient $\alpha(\theta)$ with temperature.

The other form of equation to describe the variation of α with temperature can be derived from a thermodynamical correlation and gives an equation of the form

$$\ln 10^3 \alpha = \frac{A}{T} + B \cdot \ln T + C$$
(24)

where A, B and C are constants and T is the temperature in K. For oxygen dissolved in water we find by fitting the equation to the values of α in Table 14.2 that A = 8.553 * 10³, B = 2.378 * 10, and C = -1.608 * 10².

Values of α calculated from eqns. 23 and 24 for the same temperature agree within ±0.5%.

The Bunsen absorption coefficient, however, is not a very practical measure. Values of $\alpha(\theta)$ have therefore to be converted to mg/L, and the method for doing this is best illustrated by an example.

Example: Calculation of the oxygen content $(c_S(p_{atm}, \theta))$ in air-saturated water at a temperature θ of 20°C.

Equation 24 allows the solubility of oxygen in air-saturated fresh water to be calculated for any temperature and pressure provided that the values of the Bunsen absorption coefficient $\alpha(T)$ and the vapor pressure $p_W(T)$ at the particular temperature are known. Equation 23 or 23 can be used to obtain α , and p_W can be calculated from equation 21. Table 14.3 gives oxygen solubilities in mg/L for temperature intervals of 0.1 °C from 0-40°C.

$$\mathbf{c}_{\mathrm{S}}(\mathbf{p}_{\mathrm{atm}}, \theta) = \frac{\mathbf{p}_{\mathrm{atm}} - \mathbf{p}_{\mathrm{W}}(\theta)}{\mathbf{p}_{\mathrm{N}}} \cdot 0.2095 \cdot \alpha(\theta) \cdot \frac{\mathbf{M}_{\mathrm{O}_{2}}}{\mathbf{V}_{\mathrm{M}}}$$
(25)

In equation 25 p_{atm} is the actual atmospheric pressure corrected for the contribution of the water vapor pressure p_w and related to standard pressure p_N . The corrected pressure is multiplied by 0.2095, the volume content of oxygen in air, by $\alpha(\theta)$ and by the molecular mass of oxygen (M_{O2}) divided by the molar volume V_M.

At a given atmospheric pressure of 1013 mbar ($p_{atm} = p_N$) and a temperature of 20 °C the oxygen content can be calculated according to equation 26.

$$c_{s}(1013 \text{ mbar}, 20^{\circ}\text{C}) = \frac{1013 - 23.3}{1013} \cdot 0.2095 \cdot 0.031 \cdot \frac{32 \cdot \text{g/mol}}{22.414 \text{ mol/L}} = 0.009 \text{ g/L} = 9.06 \text{ mg/L}$$
(26)

Figure 14.9 shows the temperature-dependent oxygen solubility in air-saturated fresh water and Table 14.3 gives oxygen solubilities in mg/L for temperature intervals of 0.1 $^{\circ}$ C from 0-40 $^{\circ}$ C.

Figure 12.9 Dependence of the oxygen solubility in air-saturated fresh water on temperature

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Table 1	12.3 Ox	ygen s	olubility	in air-s	aturate	d fresh	water [r	mg/L].				
T [°C]	c _s (T)	.0	.1	.2	.3	.4	.5	.6	.7	.8	.9	1.0
0	14.	64	60	55	51	47	43	39	35	31	27	23
1		23	19	15	10	06	03	99	95	91	87	83
2	13.	83	79	75	71	68	64	60	56	52	49	45
3		45	41	38	34	30	27	23	20	16	12	09
4		09	05	02	98	95	92	88	85	81	78	75
5	12.	75	71	68	65	61	58	55	52	48	45	42
6		42	39	36	32	29	26	23	20	17	14	11
7		11	08	05	02	99	96	93	90	87	84	81
8	11.	81	78	75	72	69	67	64	61	58	55	53
9		53	50	47	44	42	39	36	33	31	28	25
10		25	23	20	18	15	12	10	07	05	02	99
11	10.	99	97	94	92	89	87	84	82	79	77	75
12		75	/2	70	67	65	63	60	58	55	53	51
13		51	48	46	44	41	39	37	35	32	30	28
14		28	26	23	21	19	17	15	12	10	80	06
15	•	06	04	02	99	97	95	93	91	89	87	85
16	9.	85	83	81	70	76	74 54	72	70	68	66	64
17		64 45	62	60	58	00 07	54 25	53	51	49	47	45
10		40	43	41	39	37	30	33	31	30	28	20
19		20	24	22	20	19	17	15	13	04	09	00
20	0	00	00	04 07	02	01	99 00	97	90 70	94 76	92 75	90 72
21	0.	90 72	00 71	07 70	60 62	03 66	0Z 65	00 63	10 62	70 60	70 59	73 57
22		73 57	55	70 53	00 52	50	40 10	03 17	46	44	12	J1
23		٥ <i>٦</i>	30	38	36	35	23	32	30	28	72 27	25
25		25	24	22	21	19	18	16	15	14	12	11
26		11	09	08	06	05	03	02	00	99	98	96
27	7	96	95	93	92	90	89	88	86	85	83	82
28		82	81	79	78	77	75	74	73	71	70	69
29		69	67	66	65	63	62	61	59	58	57	55
30		55	54	53	51	50	49	48	46	45	44	42
31		42	41	40	39	37	36	35	34	32	31	30
32		30	29	28	26	25	24	23	21	20	19	18
33		18	17	15	14	13	12	11	09	08	07	06
34		06	05	04	02	01	00	99	98	97	96	94
35	6.	94	93	92	91	90	89	88	87	85	84	83
36		83	82	81	80	79	78	77	75	74	73	72
37		72	71	70	69	68	67	66	65	64	63	61
38		61	60	59	58	57	56	55	54	53	52	51
39		51	50	49	48	47	46	45	44	43	42	41
40		41	40	39	38	37	36	35	34	33	32	31

Table 12.3	Oxygen	solubility in	air-saturated	fresh wa	ater [mg/l	_].
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Example:: c_S(14.3°C) = 10.21 mg/L

12.4.3 Dependence on the Salt Concentration

Table 12.4 gives values of the concentration of dissolved oxygen at several temperatures in solutions with various chloride concentrations. Increasing the salt concentration, there is a decrease in the oxygen solubility. This behavior is characteristic for the solubility of many nonelectrolytes - it is the phenomenon known as the *salting-out effect*.

Instead of chlorinity [Cl⁻] - the amount of chloride in parts per thousand - which was used as a measure of the amount of salt in water, the term salinity is often used. If salinity is preferred as a measure of salt concentration, then the conversion from g/L can be readily made using equation 27.

$$S = 1.805[Cl^{-}] + 0.03$$

where S is the salinity in [‰] or [g/1000g]

Table 12.4 Solubility of oxygen in water as a function of temperature and salt concentration (Total pressure = 760 torr)

[Cl ⁻] (g/1000g)	0	4	8	12	16	20				
T [°C]		Oxygen solubility [mg/L]								
0	14.5	13.9	13.3	12.6	12.0	11.3				
10	11.3	10.8	10.4	9.9	9.5	9.0				
20	9.1	8.8	8.5	8.1	7.8	7.4				
30	7.5	7.3	7.0	6.7	6.4	6.1				

The effect of increasing the salt concentration on the vapor pressure is negligibly small as shown in Table 12.5.

[Cl ⁻] (g/1000g)	0	9	18	26					
T [°C]	Vapor pressure of solution (torr)								
0	4.6	4.5	4.4	4.4					
10	9.2	9.1	8.9	8.8					
20	17.5	17.3	17.0	16.7					
30	31.8	31.4	30.9	30.4					

Table 12.5. Variation of solution vapor pressure (p_W) with salt concentration

The dependence of oxygen solubility on salt concentration can also be obtained from equation 21 except that now values calculated from either equation 28 or 29 have to be used.

$$10^{3} \cdot \alpha = a + b \cdot \theta + c \cdot \theta^{2} + d \cdot \theta^{3} + e \cdot \theta^{4} - [Cl^{-}] \cdot (p + q \cdot \theta + r \cdot \theta^{2} + s \cdot \theta^{3} + t \cdot \theta^{4})$$
(28)

where θ is the temperature in °C, a - e are the coefficients used in equation 23 and p - t are new constants given in Table 12.6. The values of these new constants, obtained by fitting the polynomial to experimental data in the ranges $0 \le \theta \le 30$ °C and $0 \le [Cl^-] \le 20$ ‰. To obtain an oxygen solubility from the Bunsen absorption coefficient the same procedure as described previously is used.

An alternative equation to compensate the Bunsen absorption coefficient by the salt concentration displays equation 29.

$$10^{3} \cdot \alpha = \exp\left[\left(A + \frac{B}{T} + C \cdot \ln T + D \cdot T\right) - \left[Cl^{-}\right]\left(P + \frac{Q}{T} + R \cdot \ln T + S \cdot T\right)\right]$$
(29)

(27)

where T is the temperature in Kelvin, and A - D and P - S are the coefficients also given in Table 12.6. They are based on measurements for 273.1 \leq T \leq 308.18 K and 0 \leq [Cl⁻] \leq 30% and is therefore more extensive than equation **28**. Both equations give values of 10³ α which agree to better than \pm 1%.

Eqn. 26	а	4.900 * 10	р	5.516 * 10 ⁻¹
	b	-1.335	q	-1.759 * 10 ⁻²
	С	2.759 * 10 ⁻²	r	2.253 * 10 ⁻⁴
	d	-3.235 * 10 ⁻⁴	S	-2.654 * 10 ⁻⁷
	е	1.614 * 10 ⁻²	t	5.362 * 10 ⁻⁸
Eqn. 27	A	-7.424	Р	-1.288 * 10 ⁻¹
	В	4.417 * 10 ³	Q	5.344 * 10
	С	-2.927	R	-4.442 * 10 ⁻²
	D	4.238 * 10 ⁻²	S	7.145 * 10 ⁻⁴

Table 12.6	Values of the	coefficients in	equations 2	26 and 27.
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Seawater has a salinity typically of 35‰ (35g / 1000g) or a chloride content of about 19‰, and therefore, falls within the scope of both equations.

12.5 Temperature Compensation of the Response of Oxygen Sensors

A typical oxygen response characteristic at different temperatures is shown in Figure 12.10. The phase angle Φ is a function of the oxygen content (Φ = tan Φ = f(O₂)) and decreases with increasing the oxygen content.

Figure 12.10 Oxygen response characteristics at different temperatures.

Figure 12.11 displays the oxygen-dependence of the phase angle at different temperatures and Figure 12.12 the respective Stern-Volmer plots. These two figures and Table 12.7 displays, that both the phase angle in absence of oxygen, Φ_0 , and the Stern-Volmer constant, K_{SV}, are temperature dependent. Φ_0 decreases with increasing temperature, while KSV increases with increasing temperature.

Figure 12.11 Effect of the temperature on the phase angle at different oxygen contents given in % airsaturation.

Figure 12.12 Effect of the temperature on the Stern-Volmer constant.

Table 12.7 Effect of the temperature on the phase angle in the absence of oxygen (Φ_0) and the Stern-Volmer constant K_{SV}.

θ [°C]	3.5	10	20	30	40
$\Phi_0 [^\circ]$	61.88	61.23	60.38	59.48	58.48
K _{SV} ^a [% air-sat] ⁻¹	0.05018	0.05429	0.05887	0.06384	0.06924

^a: The Stern-Volmer constant is determined via equation 9.

Figure 12.13 displays the temperature dependence of Φ_0 and K_{SV}. From Table 12.7 and Figure 12.13 a decreases in Φ_0 of about 0.09° can be calculated by increasing the temperature by 1 K. On the other hand, the Stern-Volmer constant (K_{SV}) increases about 5.0*10⁻⁴ [% air-sat.]⁻¹ by increasing the temperature by 1 K.

Figure 12.13 Effect of the temperature on Φ_0 and the Stern-Volmer constant (K_{SV}).

Consequently, variations in the temperature causes variations in the measuring value at a constant oxygen content (see Figure 12.11). Table 12.8 displays the deviation of the measuring value from the real oxygen content depending on temperature and the oxygen content.

Table 12.8 Variation of the measured oxygen content (Δ % air-saturation) at a constant oxygen content and variations in temperature by ±1K.

% air-saturation	25 % air-sat.	100 % air-sat.	250 % air-sat.
	(50.9 hPa)	(203.6 hPa)	(508.9 hPa)
Δ %air-saturation /K	$\pm 0.43 \ (0.88 \text{ hPa})$	± 1.62 (3.30 hPa)	± 3.91 (7.96 hPa)

Example:

If the temperature is measured with a precision of \pm 0.2 °C, there is a variation in the measuring value at 100% air-saturation of 100 \pm 0.7 % air-saturation.

WARRANTY

WPI (World Precision Instruments) warrants to the original purchaser that this equipment, including its components and parts, shall be free from defects in material and workmanship for a period of one year* from the date of receipt. WPI's obligation under this warranty shall be limited to repair or replacement, at WPI's option, of the equipment or defective components or parts upon receipt thereof f.o.b. WPI, Sarasota, Florida U.S.A. Return of a repaired instrument shall be f.o.b. Sarasota.

The above warranty is contingent upon normal usage and does not cover products which have been modified without WPI's approval or which have been subjected to unusual physical or electrical stress or on which the original identification marks have been removed or altered. The above warranty will not apply if adjustment, repair or parts replacement is required because of accident, neglect, misuse, failure of electric power, air conditioning, humidity control, or causes other than normal and ordinary usage.

To the extent that any of its equipment is furnished by a manufacturer other than WPI, the foregoing warranty shall be applicable only to the extent of the warranty furnished by such other manufacturer. This warranty will not apply to appearance terms, such as knobs, handles, dials or the like.

WPI makes no warranty of any kind, express or implied or statutory, including without limitation any warranties of merchantability and/or fitness for a particular purpose. WPI shall not be liable for any damages, whether direct, indirect, special or consequential arising from a failure of this product to operate in the manner desired by the user. WPI shall not be liable for any damage to data or property that may be caused directly or indirectly by use of this product.

Claims and Returns

Inspect all shipments upon receipt. Missing cartons or obvious damage to cartons should be noted on the delivery receipt before signing. Concealed loss or damage should be reported at once to the carrier and an inspection requested. All claims for shortage or damage must be made within ten (10) days after receipt of shipment. Claims for lost shipments must be made within thirty (30) days of receipt of invoice or other notification of shipment. Please save damaged or pilfered cartons until claim is settled. In some instances, photographic documentation may be required. Some items are time-sensitive; WPI assumes no extended warranty or any liability for use beyond the date specified on the container

Do not return any goods to us without obtaining prior approval and instructions from our Returns Department. Goods returned (unauthorized) by collect freight may be refused. Goods accepted for restocking will be exchanged or credited to your WPI account. Goods returned which were ordered by customers in error are subject to a 25% restocking charge. Equipment which was built as a special order cannot be returned.

Repairs

Contact our Customer Service Department for assistance in the repair of apparatus. Do not return goods until instructions have been received. Returned items must be securely packed to prevent further damage in transit. The Customer is responsible for paying shipping expenses, including adequate insurance on all items returned for repairs. Identification of the item(s) by model number, name, as well as complete description of the difficulties experienced should be written on the repair purchase order and on a tag attached to the item.

* Electrodes, batteries and other consumable parts are warranted for 30 days only from the date on which the customer receives these items.

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