SENSOR PROBES

Oxygen Dipping Probe PSt3 / PSt6 / PSt9

Instruction Manual

PreSens
PRECISION SENSING

Oxygen Dipping Probe

PSt3 / PSt6 / PSt9

Specification:

Oxygen dipping probe with high grade stainless steel fitting for normal / trace / ultra-low oxygen range

Document filename: IM_DP-PSt3_PSt6_PSt9_dv2

Specifications may change without prior notice.

Manufacturer
PreSens Precision Sensing GmbH
Am BioPark 11
93053 Regensburg, Germany
Phone +49 941 94272100
Fax +49 941 94272111
info@PreSens.de
www.PreSens.de

Table of Contents

1 Preface

You have chosen a new, innovative technology for measuring oxygen.

Chemical optical sensors (also called optodes) have several important features:

- They are small.
- Their signal does not depend on the flow rate of the sample.
- They can be physically divided from the measuring system which allows a non-invasive measurement.
- They can be used in disposables.

Therefore, they are ideally suited for the examination of small sample volumes, for highly parallelized measurements in disposables, and for biotechnological applications.
A set of different minisensors, flow-through cells and integrated sensor systems is available to make sure you have the sensor which matches your application.

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

Your PreSens Team

PLEASE READ THE FOLLOWING INSTRUCTIONS CAREFULLY BEFORE WORKING WITH THIS ITEM.

2 Safety Notes

! It is the customer’s responsibility to validate the sensor under end-user conditions according to safety precautions of the application to ensure that the use of the sensor is safe and suitable for the intended purpose.

PreSens is explicitly not liable for direct or indirect losses caused by the application of these sensors. In particular it has to be considered that malfunctions can occur due to the naturally limited lifetime of the sensor depending on the respective application. The set-up of backup measurement stations is recommended when using the sensors in critical applications to avoid consequential losses. It is the customer’s responsibility to install a suitable safety system in the event of sensor failure.

3 Description of the Oxygen Dipping Probe

Oxygen dipping probes measure the partial pressure of both gaseous and dissolved oxygen. They consist of a polymer optical fiber (POF) with a polished distal tip which is coated with a planar oxygen sensitive foil. Oxygen dipping probes are available with sensor coatings type PSt3 (detection limit 15 ppb, 0 - 100 % oxygen), type PSt6 (detection limit 1 ppb, 0 - 4.2 % oxygen), and PSt9 (detection limit 0.5 ppm, 0 - 200 ppm). The end of the polymer optical fiber is covered with a high-grade steel tube to protect both the sensor material and the POF. The cable has an outer diameter of 2.8 mm. The inner diameter of the POF is 2.0 mm. The steel tube has an outer diameter of 4 mm. Usually the fiber is coated with an optical isolated sensor material in order to exclude ambient light from the fiber tip and to increase chemical resistance especially against oily samples as well as to reduce biofouling on the sensor membrane. This type of oxygen sensor has excellent long-term stability.

Fig. 1 Oxygen dipping probe; insert: enlarged view of sensor coating

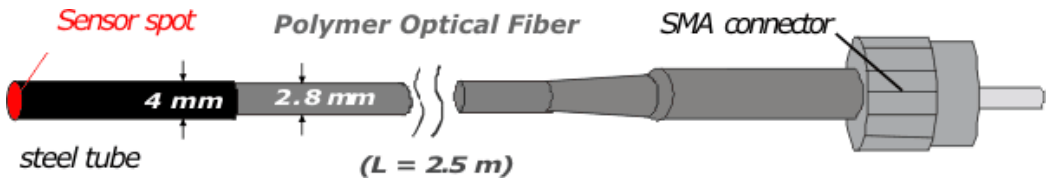

Fig. 2 Schematic illustration of an oxygen dipping probe

Dipping probe features:

- No polarization necessary
- No membrane cleaning and replacement necessary
- No electrolyte solutions to poison or replenish
- Long shelf-life
- Pressure resistant up to 3 bar

3.1 Scope of Delivery

The oxygen dipping probe is delivered with a protective rubber cap on the distal end of the steel fitting to protect the sensor coating.

! Please remove the protective rubber cap before inserting the dipping probe in your sample.

Additionally required equipment (not supplied):

- Fiber optic oxygen transmitter Fibox 4 / Fibox 4 trace (more oxygen transmitters can be found on www.presens.de/products/o2/meters.html)
- PC / Notebook

3.2 Measurement Set-up

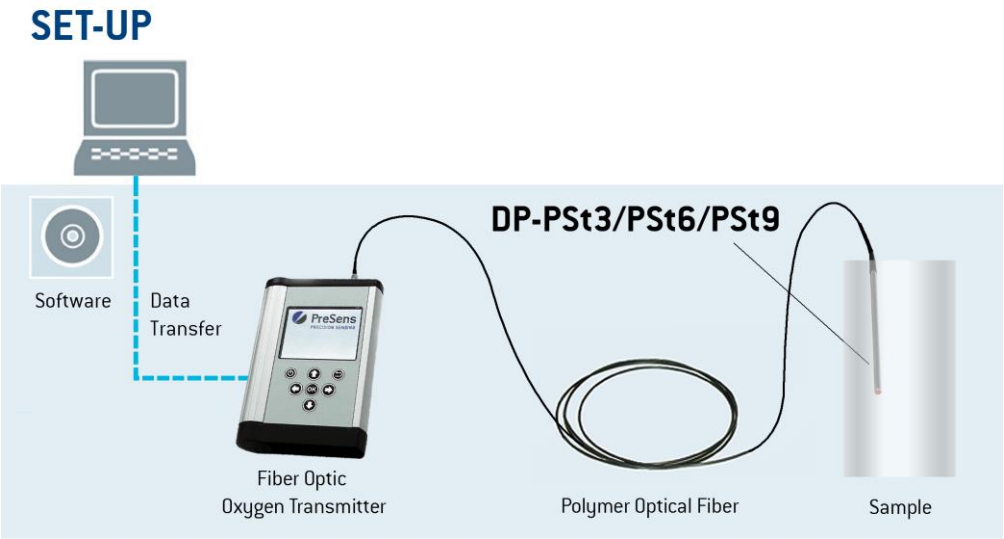

Fig. 3 Set-up for measurement with an oxygen dipping probe

Connect the dipping probe's polymer optical fiber to the sensor connector of the fiber optic oxygen meter (e. g. Fibox 4 trace). Then remove the protective rubber cap from the steel fitting and insert the dipping probe in your sample.

3.2.1 Underwater Applications

For underwater applications specially designed weights for dipping probes are available, which keep the probe in a stable, fixed position while it is submerged. For more information please see the Sensor Probe Accessories instruction manual (IM_SP_Acc).

Fig. 4 Dipping Probe with attached DP-Weight for under water use.

3.2.2 Gas Phase Applications

For applications in the gas phase, especially when working with gas pipelines the oxygen dipping probe can be integrated in a Swagelok T-cell. The metal T-connector with oxygen probe (FTCM) can easily be integrate in pipes with o. d. 6 mm (or other diameter, when using the respective adapter). Gases (or liquids) can be pumped through the cell and precise oxygen measurements conducted.

Fig. 5 Dipping probe (top) integrated in Swagelok T-cell (FTCM)

4 Operation

! Please note that oxygen dipping probes (DP) are NOT autoclavable – unlike the oxygen probes for inline measurements (OIM) – as the polymer optical fiber does not stand high temperatures.

Maximum temperature for a standard dipping probe is 80 °C. However, a high temperature version is available which stands temperatures up to 110 °C. In case the dipping probe must be exposed to temperatures > 80 °C please contact our service team.

4.1 Calibration

The oxygen dipping probe has to be calibrated before use. A conventional two-point calibration in oxygen-free environment (nitrogen / CO_2, or sodium sulfite), and air saturated (sensor type PSt3), 1 – 2 % oxygen (sensor type PSt6), or 100 – 200 ppmv oxygen (sensor type PSt9) environment has to be performed.
A recalibration is recommended after 100,000 measurement points or a period of 3 months, whichever is reached earlier.

Please see the respective transmitter instruction manual for more detailed information about software settings and calibration procedure.

4.1.1 Calibration of a DP-PSt3 – Preparation of Calibration Standards

1st Calibration Point:

- **In Liquid:** Oxygen-free water
  To prepare oxygen-free water dissolve 1 g of sodium sulfite (Na_2SO_3) and 50 µL cobalt nitrate ($Co(NO_3)_2$) standard solution (ρ(Co) = 1000 mg/L; in nitric acid 0.5 mol/L) in 100 mL water. Use a suitable vessel with a tightly fitting screw top and label it **cal 0**. Make sure there is only little headspace in your vessel. Due to a chemical reaction of oxygen with the Na_2SO_3 the water becomes oxygen-free. Additional oxygen, diffusing from air into the water, is removed by surplus Na_2SO_3. Close the vessel with the screw top and shake it for approximately one minute to dissolve Na_2SO_3 and to ensure that the water is oxygen-free. To prepare oxygen-free water you also can use sodium dithionite ($Na_2S_2O_4$).

  Place the oxygen dipping probe in the vessel with **cal 0**. To minimize the response time, slightly stir the solution. Then follow the instructions in the respective transmitter manual for calibration. After recording the first calibration point remove the dipping

probe from the calibration solution **cal 0**, and rinse it with distilled water. Repeat this procedure at least 5 times to clean the probe from sodium sulfite.

For storing the calibration solution **cal 0** keep the vessel closed after calibration with a screw top to minimize oxygen contamination. The shelf life of **cal 0** is about 24 hours provided that the vessel has been closed with the screw top.

- **In Humid Gases**: Nitrogen-saturated atmosphere
  Use nitrogen-saturated atmosphere as calibration standard **cal 0**. Use a commercially available test gas N_2(5.0) (suppliers are e. g. Air Liquide, Linde Westfalen AG). Lead the gas into a vessel filled with distilled water before feeding it into the calibration vessel with the dipping probe (see Fig. 6). Then follow the instructions in the respective transmitter manual.

- **In Dry Gases**: Nitrogen-saturated atmosphere
  Use nitrogen-saturated atmosphere as calibration standard **cal 0**. Use a certified test gas N_2(5.0) (suppliers are e. g. Air Liquide, Linde Westfalen AG). Lead the gas directly into the calibration vessel with the dipping probe in it (see Fig. 7). Then follow the instructions in the respective transmitter manual.

2nd Calibration Point:

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

  Place the oxygen dipping probe in the vessel with **cal 100**. To minimize the response time, slightly stir the solution. Then follow the instructions in the respective transmitter manual for calibration.

- **In Humid Gases**: Water vapor-saturated air or 20.9 % O_2
  Use water-vapor saturated air as calibration standard **cal 100**. Place wet cotton wool in a vessel with a fitting screw top or lid.

  For inserting dipping probe (and the temperature sensor) into the vessel you might have to drill holes in the lid. Introduce the dipping probe and wait about 2 minutes to ensure that the air is water vapor-saturated.

  Alternatively, you can use a certified test gas O_2(20.9) (suppliers are e. g. Air Liquide, Linde Westfalen AG). Lead the gas into a vessel filled with distilled water before

feeding it into the calibration vessel with the dipping probe (see Fig. 6). Then follow the instructions in the respective transmitter manual for calibration.

- **In Dry Gases**: 20.9 % O_2
  Leave the dipping probe in ambient air (or certified test gas O_2(20.9); suppliers are e. g. Air Liquide, Linde Westfalen AG) . Then follow the instructions in the respective transmitter manual.

4.1.2 Calibration of a DP-PSt6 – Preparation of Calibration Standards

Calibration of a DP-PSt6 is performed with certified gases (suppliers are e. g. Air Liquide, Linde, Westfalen AG).

1st Calibration Point:

- **In Humid Gases**: Nitrogen- or CO_2-saturated atmosphere
  Use nitrogen or CO_2 (N_2 / CO_2 5.0) as a first calibration standard **cal 0**.
  Lead the gas into a vessel filled with distilled water before feeding it into the calibration vessel with the dipping probe (see Fig. 6). Then follow the instructions in the respective transmitter manual.

- **In Dry Gases**: Nitrogen- or CO_2-saturated atmosphere
  Use nitrogen or CO_2 (N_2 / CO_2 5.0) as a first calibration standard **cal 0**.
  Lead the gas directly into the calibration vessel with the dipping probe in it (see Fig. 7). Then follow the instructions in the respective transmitter manual.

2nd Calibration Point:

- **In Humid Gases**: 1 – 2 % O_2 (humidified gas)
  The second calibration value **cal 2nd** for a PSt6 sensor is ideally in the range between 5 and 10 % air sat. (ca. 1 – 2 % O_2 → % air sat. = % O_2 x 100/20.95). Use a commercially available test gas of 1 – 2 % O_2 as a second calibration standard **cal 2nd**.
  Lead the gas into a vessel filled with distilled water before feeding it into the calibration vessel with the dipping probe (see Fig. 6). Then follow the instructions in the respective transmitter manual.

- **In Dry Gases**: 1 – 2 % O_2
  Use a commercially available test gas of 1 – 2 % O_2 as a second calibration standard **cal 2nd**.
  Lead the gas directly into the calibration vessel with the dipping probe in it (see Fig. 7). Then follow the instructions in the respective transmitter manual.

If it is not possible to use gases or to build a suitable calibration chamber, a manual calibration can be performed, using calibration values obtained from the final inspection protocol delivered with the dipping probe. You can find more information about manual calibration in the instruction manual of the respective transmitter.

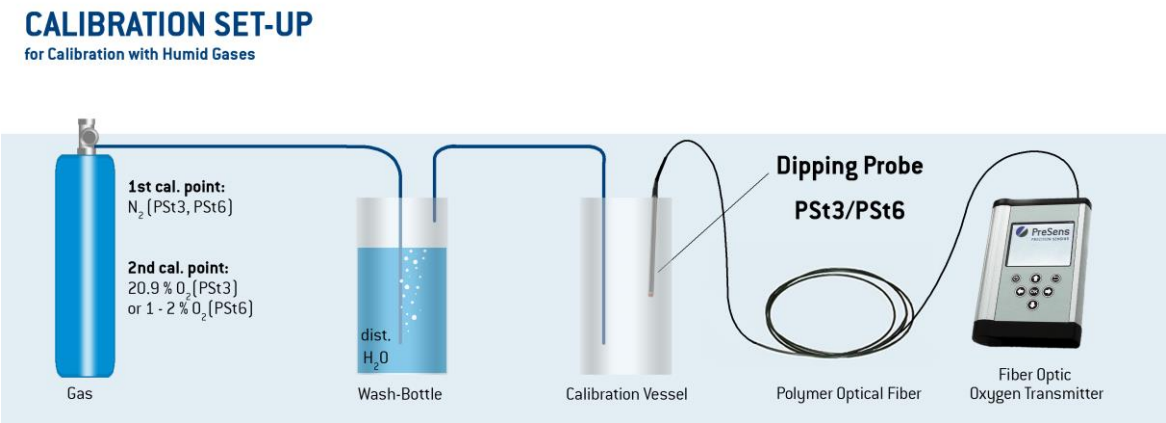

Fig. 6 Calibration set-up: Two-point calibration of a dipping probe with humid gases

4.1.3 Calibration of a DP-PSt9 – Preparation of Calibration Standards

A PSt9 sensor can only be calibrated with dry gases. Calibration is performed with certified gases (suppliers are e. g. Air Liquide, Linde, Westfalen AG).

1st Calibration Point:

- Use nitrogen-saturated atmosphere as calibration standard **cal 0**. Use a certified test gas N_2(6.0), and lead the gas directly into the calibration vessel with the dipping probe in it (see Fig. 7). Then follow the instructions in the respective transmitter manual.

2nd Calibration Point:

- Use a certified test gas of 100 – 200 ppmv O_2 as calibration standard **cal 2nd**, and lead the gas directly into the calibration vessel with the dipping probe in. Then follow the instructions in the respective transmitter manual.

If it is not possible to use gases or to build a suitable calibration chamber, a manual calibration can be performed, using calibration values obtained from the final inspection

protocol delivered with the dipping probe. You can find more information about manual calibration in the instruction manual of the respective transmitter.

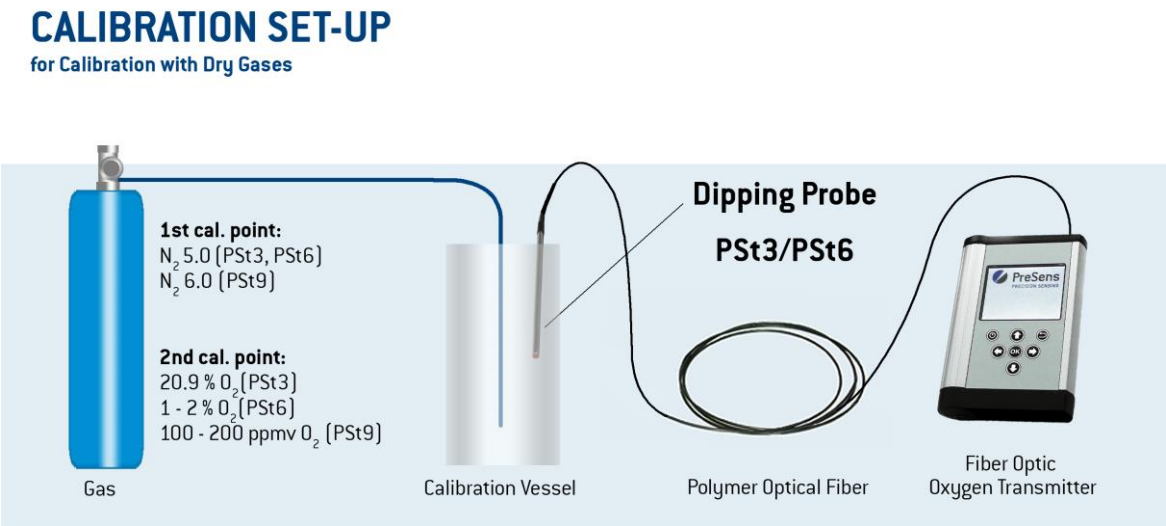

Fig. 7 Calibration set-up: Two-point calibration of a dipping probe with dry gases

4.2 Performance Proof

If you want to prove the sensor performance during the past measurement, please check the calibration values by inserting the sensor in the cal0 and cal2nd calibration standards (see chapter 3.1) when you have finished your measurement. If the device shows 0% air saturation immersing the sensor tip into cal0 and the respective oxygen concentration for the cal2nd standard, the sensor worked perfectly throughout the whole measurement.

5 Technical Data

	PSt3		PSt6		PSt9
Specifications	**Gaseous & Dissolved O_2**	**Dissolved O_2**	**Gaseous & Dissolved O_2**	**Dissolved O_2**	**Gaseous O_2**
Measurement range	0 – 100 % O_2 0 – 1000 hPa	0 – 45 mg/L 0 – 1400 µmol/L	0 – 4.2 % O_2 0 – 41.4 µmol/L	0 – 1.8 mg/L 0 – 56.9 µmol/L	0 – 200 ppm
Limit of detection	0.03 % O_2	15 ppb	0.002 % O_2	1 ppb	0.5 ppm
Resolution	± 0.01 % O_2 at 0.21 % O_2 ± 0.1 % O_2 at 20.9 % O_2 ± 0.1 hPa at 2 hPa ± 1 hPa at 207 hPa	± 0.14 µmol/L at 2.83 µmol/L ± 1.4 µmol/L at 238.1 µmol/L	± 0.0007 % O_2 at 0.002 % O_2 ± 0.0015 % O_2 at 0.2 % O_2 ± 0.007 hPa at 0.023 hPa ± 0.015 hPa at 2.0 hPa	± 0.010 µmol/L at 0.03 µmol/L ± 0.020 µmol/L at 2.8 µmol/L	10 ± 0.5 ppm 100 ± 0.8 ppm 200 ± 1.5 ppm
Accuracy	± 0.4 % O_2 at 20.9 % O_2 ± 0.05 % O_2 at 0.2 % O_2		± 1 ppb or ± 3 %; whichever is higher		± 2 ppm or ± 5 %; whichever is higher
Measurement temperature range	0 to +50 °C		0 to +50 °C		0 to +40 °C
Response time (t_{90})	< 6 sec.	< 40 sec.	< 6 sec.	< 40 sec.	< 3 sec.
Properties					
Compatibility	Aqueous solutions, ethanol, methanol				Gas phase only
No cross-sensitivity with	pH 1 – 14 CO_2, H_2S, SO_2 Ionic species				CO_2, SO_2
Cross-sensitivity	Organic solvents, such as acetone, toluene, chloroform or methylene chloride Chlorine gas				Organic vapor, Chlorine gas
Sterilization procedure	Ethylene oxide (EtO) Gamma irradiation				-
Cleaning procedure	3 % H_2O_2 Acidic agents (HCl, H_2SO_4), max. 4 – 5 % at room temperature				-
Calibration	Two-point calibration with oxygen-free environment (nitrogen, sodium sulfite) and air-saturated environment		Two-point calibration in oxygen-free environment (nitrogen) and a second calibration value optimally between 1 and 2 % oxygen		Two-point calibration in oxygen-free environment (nitrogen 6.0) and a second calibration value optimally between 100 and 200 ppm gaseous oxygen
Storage Stability	2 years provided the sensor material is stored in the dark (-10 to +60 °C)				

6 Concluding Remarks

Dear Customer,

With this manual, we hope to provide you with an introduction to work with the oxygen dipping probes type PSt3, PSt6 and PSt9.

This manual does not claim to be complete. We are endeavored to improve and supplement this version.

We are looking forward to your critical review and to any suggestions you may have.

You can find the latest version at www.PreSens.de.

With best regards,

Your PreSens Team

Manufacturer

PreSens
Precision Sensing GmbH

Am BioPark 11
93053 Regensburg
Germany

Phone +49 941 94272100
Fax +49 941 94272111

info@PreSens.de
www.PreSens.de