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O2k-Core Manual B. Oxygraph-2k Series E-F, DatLab 5.1

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Service of the Polarographic Oxygen Sensor OroboPOS

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Summary: Service of the polarographic oxygen sensors (OroboPOS) is the basis for signal stability, low noise and high time resolution. Sensor service may not be required for several months of operation. Performance of the OROBOROS Oxygraph-2k according to instrumental specifications is obtained only with oxygen sensors which are maintained in a good functional state.

POS service is required if (1) the oxygen signal does not decline rapidly to a low level of zero current (0% to max. 5%) at zero oxygen pressure, (2) the time response is prolonged (time constant >10 s) and biphasic, (3) signal noise increased, or (4) the raw signal at air calibrations declines. For each sensor, the frequency of POS service can be optimized on the basis of a long-term record of calibration values.

1. Accessories

Accessories for sensor service are provided in the OroboPOS-Service Kit (Fig. 1). In addition, you need distilled water and 25% ammonia solution (fresh).

Fig. 1. **OroboPOS-Service Kit - Oxygraph-2k**

①	26300-01	OroboPOS-Electrolyte powder, KCl
②	26200-01	OroboPOS-Membranes, FEP 25 µm; 40/Pck.
③	26520-01	OroboPOS-Polishing Powder for cathode cleaning
④	26510-01	OroboPOS-Polishing Cloth for cathode cleaning
⑤	26400-01	OroboPOS-Mounting Tool for membrane application
⑥	26800-01	Pipette\Plastic\1 ml ungraded for electrolyte
⑦	26600-01	O2-Zero Powder, dithionite ($Na_2S_2O_4$)
	26550-01	Pen-Contact Oil for stable low contact resistance

2. Cleaning

Observe the guidelines for preventing damage by electrostatic discharge (ESD) when handling the POS connectors or the cable connections to the O2k (MiPNet14.01).

During cleaning of the anode and cathode, the sensor is best mounted to the blue POM base of the perspex vial in which the OroboPOS sensor is shipped. Alternatively, it can be mounted to the OroboPOS-Connector.

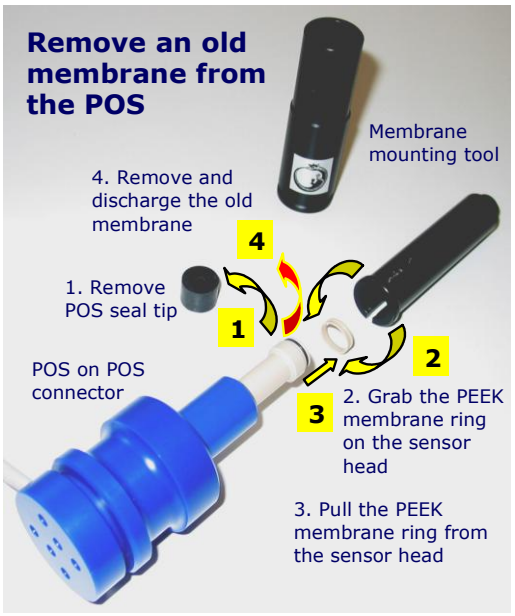

Figure 2. Removal of a used membrane from the POS.

2.1. Anode cleaning

The silver/silver chloride anode darkens after long-term operation, contact with hydrogen sulfide or inadequate storage of the sensor. This may cause high signal noise or reduces the signal output by up to >30%, reflected by the requirement to increase the gain. Such a sensor can be improved by chemically cleaning the anode, with the POS head attached to the POS connector or to the blue base of the Perspex housing of the POS (OroboPOS-Service kit). If necessary, clean the cathode first (Section 2.2). Remove the old membrane (Fig. 2). Wash off the electrolyte with distilled water, and fill the electrolyte reservoir of the sensor with concentrated (25%) ammonia solution. After up to 10 minutes the silver/silver chloride should appear bright gray. Wash the sensor carefully with distilled water. Repeat the application of ammonia solution twice. With severely aged sensors it might be necessary to prolong the exposure to ammonia up to several hours (overnight), sealing the ammonia under a membrane and under the POS cover slip in long-term applications. Protect the POS from light, since the silver/silver chloride anode is light sensitive.

2.2. Cathode Cleaning

The cathode must be treated with extreme care. The cathode must not be touched with the fingers, nor exposed to detergents or greasy liquids.

The cathode should be cleaned only when necessary, for instance when its golden surface appears to be coated by a colored layer. Wash off the electrolyte with distilled water. Then clean the cathode by polishing, using fine aluminum oxide powder (0.3 μm) and the polishing cloth supplied in the POS recharge kit (Fig. 1). Place the Petri dish with the cloth on a flat surface, add some grinding powder with the tip of a spatula, and moisten it with a few drops of distilled water to obtain a thin paste (Fig. 3). Hol the sensor upright and polish the cathode for one minute in a figure-eight motion (Fig. 3). Wash the grinding powder carefully off the sensor with distilled water, and wash the cleaning tools as well.

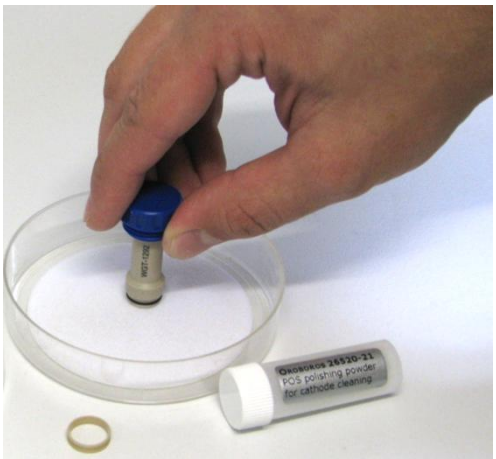

Figure 3. Cathode cleaning.

The response time of the sensor signal may remain high with a biphasic pattern (exponential phase followed by a slow drift) even after polishing and cleaning with ammonia. You may further clean the gold cathode with ultrasonic treatment at low power for up to 30 seconds while immersing the tip of the sensor head in distilled water. In extreme and rare cases, the cathode may be cleaned by adding a drop of 50-75% nitric acid onto the surface of the cathode for only 15 seconds (no longer) with care.

Remove carefully any traces of nitric acid by washing with distilled water, and proceed as described above. After cleaning the cathode, the anode must be cleaned as well (Section 2.1).

2.3. Cleaning of the Electrical Connection

Unscrew the POS head and inspect both sides of the electrical connection (gold pin and threads). Remove any contamination such as grease and moisture with a fine paper cloth. If necessary, wash with distilled water and then with pure alcohol, and apply the contact oil from the OroboPOS-Service kit to the connections. Before screwing the POS head onto the POS connector used for membrane application, check the POS connector for moisture and any other contamination (particularly for salt crystals from the electrolyte). Similarly, clean the electrical cable connection to the O2k-Main Unit (POS).

3. Membrane Mounting

After a sensor has been used and the seal tip has been removed from the sensor, it is normal to see many small bubbles. This does not indicate that there was a problem while the sensor was actually in use.

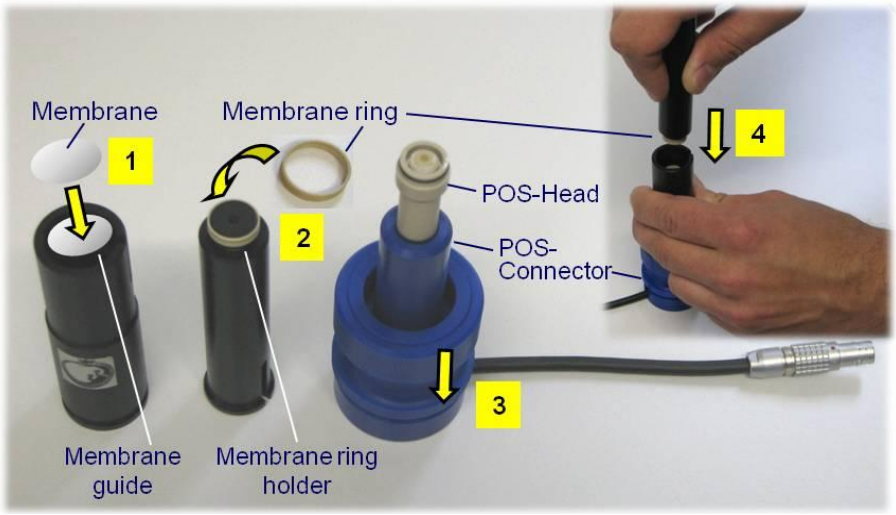

Figure 4. Mounting of a membrane onto the POS, using the membrane mounting tool which consists of two parts, the membrane guide and the membrane ring holder.

For mounting a new membrane, you may leave the POS head screwed on the sensor connector and use the mounting tool provided in the OroboPOS service kit (Fig. 4).

Before use, rinse the mounting tool with distilled water to wash off any electrolyte crystals.

The cathode and anode surfaces must be clean for a proper long-term function of the polarographic oxygen sensor (see Section 2). For membrane mounting, follow the step-by-step instructions below (Fig. 4).

Check the O-ring on the POS head to ensure that it fits properly and its surface is smooth and intact. You may apply a minuscule amount of grease to the O-ring of the sensor head (Fig.2) before adding the electrolyte.

As electrolyte, use KCl solution (1 mol·dm^{-3}; 74.56 g potassium chloride per litre, in distilled water). Add distilled water to the electrolyte powder (Fig. 1) up to the 10 ml mark. Alternatively, dissolve 1.49 g KCl in distilled water with a total volume of 20 ml. Store at room temperature or 4 °C in a closed bottle. To prevent the formation of gas bubbles in the electrolyte, heat the electrolyte solution by shaking the sealed electrolyte container in hot water (40-70 °C) before filling the electrolyte reservoir of the POS.

Add a new membrane into the membrane guide (Fig. 4; Step 1). The membranes are contained in a small box (Fig. 1, item 6). They are fully transparent, and each membrane is separated by a non-transparent paper sheet. Do not add the paper to the oxygen sensor. Separate the membrane from the paper sheets, carefully avoiding any mechanical damage of the transparent membrane. Do not touch the central area of the membrane with your fingers. The pair of forceps is particularly useful for separating the membrane and positioning it on the membrane guide of the POS mounting tool.

Fix the PEEK membrane ring (which seales the membrane against the sensor body) to the membrane ring holder (Fig. 4; Step 2). With the POS head filled with electrolyte, slide the membrane guide down the POS connector, and move the lower ring on the POS connector down to bring the membrane guide into a fixed position (Fig. 4; Step 3). Then slide the membrane ring holder into the membrane guide and press firmly down to slide the PEEK membrane ring over the POS head (Fig. 4; Step 4).

No gas bubbles should be trapped in the electrolyte reservoir after membrane application. As a check, you may inspect the electrolyte reservoir under a binocular. No folds should be visible in the membrane in the central area. Small folds in the membrane near the outer circumference have no influence, but large folds should be avoided. Wash excess electrolyte off the outside of the sensor and POS connector and wipe dry, before attaching the POS connector to the POS holder at the O2k-Main Unit.

After sensor service or membrane mounting, the POS needs some time in operation to stabilize while the O2k is switched on. Such stabilization may require several hours, and for this purpose the O2k may be left on overnight.

4. Electrical Cable Connection: O2k-Series D to F

Connect the POS cable to the O2k-Main Unit, avoiding bending and torsions of the cable. Insert the male plug of the POS cable into the female connector next to the control light of the stirrer. The red dot on the male plug has to face accurately upwards when inserting the plug.

See Appendix for O2k Series B to C.

5. Storage of the OroboPOS

5.1. Short-Term Storage in the Oxygraph-2k

For short periods of days or several weeks, the POS can be stored in the Oxygraph-2k chamber. The chamber must be washed with distilled water, and can be stored for chemical sterilization with 70% ethanol. The chamber is completely filled with 70% ethanol, and the stopper is loosely inserted. For this, do not push the stopper downwards beyond the point where the sealing ring is inserted into the glass chamber to ensure a longer life time of the sealing rings. The receptacle of the stopper may be completely filled with ethanol from the top, and is sealed with a Perspex cover slip to avoid evaporation of the ethanol. Before an experiment, the ethanol is siphoned off and the chamber is washed with distilled water. Storage with 70% ethanol is superior to storage with distilled water or electrolyte, since no subsequent chemical sterilization is required (MiPNet06.03).

5.2. Short-Term Shelf Storage

For shelf storage, the POS-Connector is unplugged from the O2k-Main Unit. Clean the sealing tip and membrane with distilled water. While the unscrewed POS connectors should be stored dry, the POS head should remain moistened by applying a cup on the sensor head to **prevent the drying out of the electrolyte**.

5.3. Long-Term Storage

For storage of the POS for several months, the sealing tip, membrane and electrolyte should be removed and the sensor **stored dry and in the dark**. Even if the membrane is not damaged, remove it by gripping the membrane holding ring with the groove in the lower end of the membrane ring holder of the mounting tool (Figure 2). Dry and dark storage of the POS prolongs its lifetime.

Wash all the electrolyte off the POS with distilled water. Check for any moisture and salt contamination in the electrical connector of the POS head. In this case, wash with distilled water and subsequently with pure methanol, dry at 60 °C for 24 h. Do not slip the membrane holder ring over the POS head during long term storage**. Store dry and dark, preferably in a container dried by silica gel.**

6. Replacement of the OroboPOS Head

After cleaning and drying the gold cathode contact or drying the sensor body at 60 °C for a few days, the zero current should be reduced. In addition, the zero current of the bare cable, without sensor head connected, is tested for any leak currents. If the latter test excludes any

sources of leak currents other than the POS, and POS service is not successful, the sensor head must be replaced.

A new sensor head can be screwed onto the sensor connector, if the old sensor head has been irreversibly damaged or should be replaced without immediate service.

7. References

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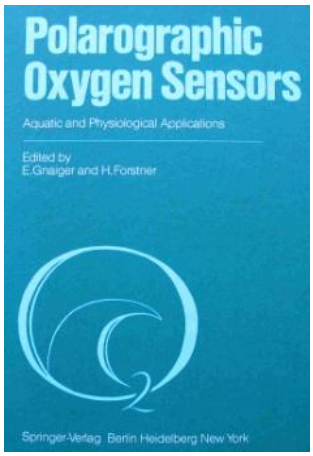
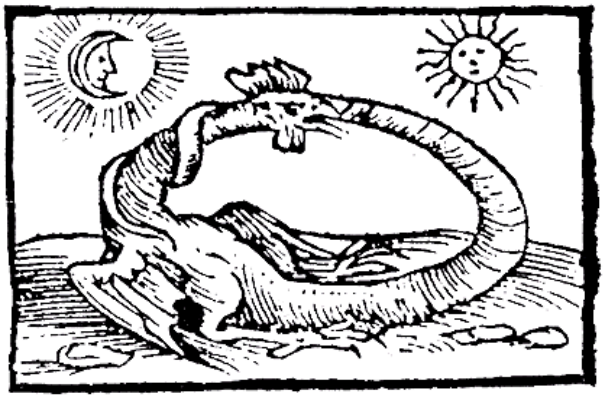
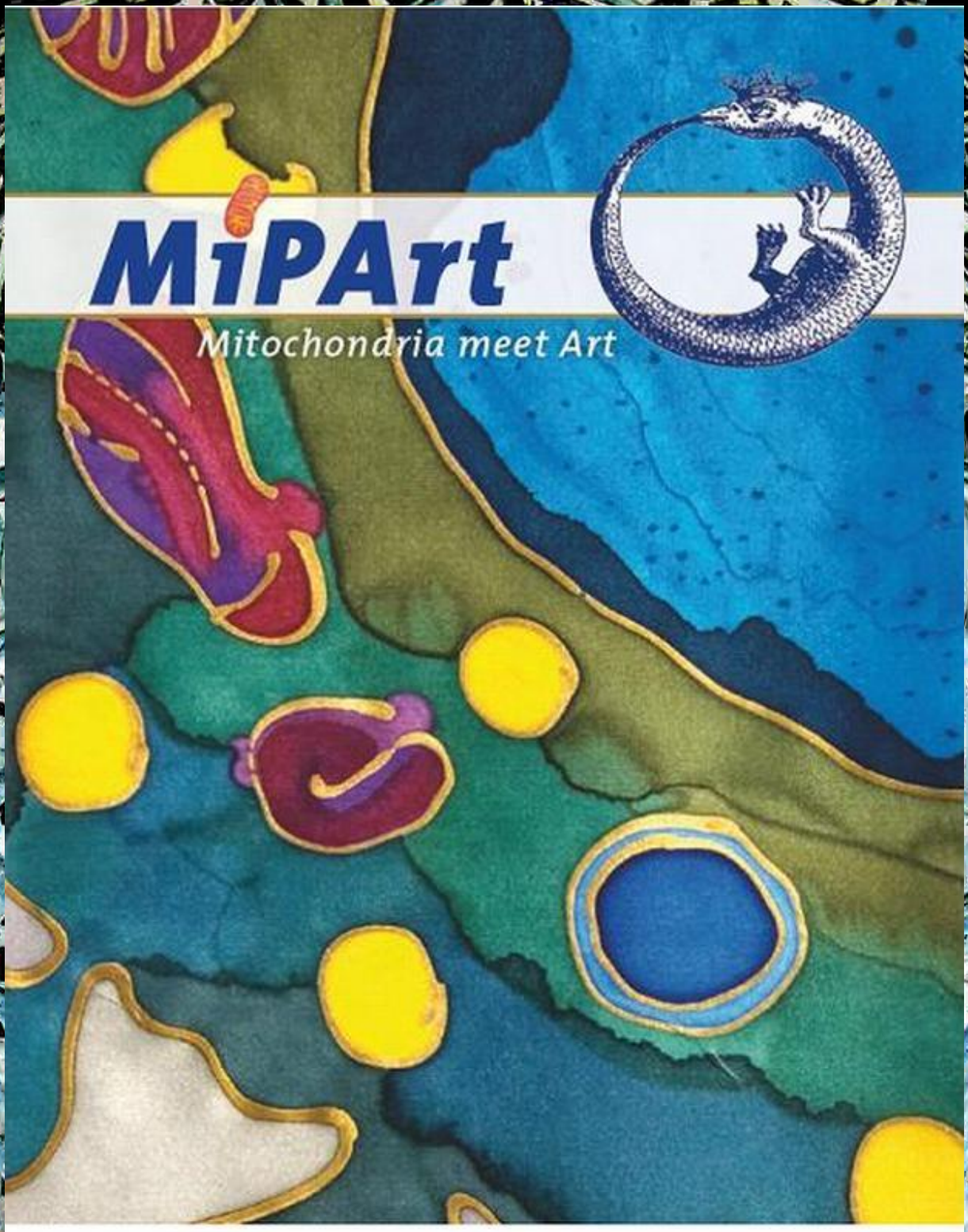


O2k-Manual Electrostatic discharge (ESD): Damage and protection. MiPNet14.01

O2k-Protocols Oxygen Calibration and solubility in experimental media. MiPNet06.03





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Appendix: O2k-Series B to C

Electrical Cable Connection: O2k-Series B to C

Insert the male plug of the POS cable into the female plug positioned between the two control lights of the Oxygraph-2k (left chamber: between the MAINS and STIRRER lights; right chamber: between the STIRRER and COMM lights). The ridge on the male plug has to face accurately downwards when inserting the plug, before rotating the connector screw and fixing it finger tight.