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1 ZOSHBI

ZOSHBI is a zirconia oxide oxygen sensor for high temperatures in ProboStat. The sensor is used in the mounted on the inner chamber gas in line. ZOSHBI has higher temperature range (500-1400°C) than our MOSE (500-1050°C) oxygen sensors.

The yttria stabilized zirconia ceramic tube is (nearly) pure oxide ion conductor at elevated temperatures. A voltmeter connected to the electrodes on both the inside and the outside surfaces of the tube can be used to measure the potential across the tube. The potential of oxygen ions across the tube can be measured from the proportional potential of electrons via the connected electrodes with a voltmeter.

This oxygen sensor construct requires a reference gas which is both supplied and evacuated from the same "Inner In" gas connection by utilizing 1/32" gas lines threaded into the 1/8" default gas line. This is a non-standard approach that requires replacing the quick connect with a bulkhead and threading a thin gas tube inside another gas tube. At this time, this product and this manual must be considered experimental without the normal assumptions of ease of use, ease of setup, warranty etc.

For oxygen sensors without the need for reference gas ask us about "MOSE".

2 Reference flush gas line

To use ZOSHBI the "inner in" gas connection needs to be converted to a system that is able to both send and receive the reference gas. This is done by threading 1/32" gas line inside the 1/8" gas line.

Such arrangement does not change the functionality of the Probostat, but nevertheless can removed at any time by pulling the RG line out from the bulkhead.

2.1 New ProboStat

In case a new ProboStat is purchased same time as ZOSHBI the reference gas (RG) flush line and the adapter will be preinstalled.

2.2 Existing ProboStat

In this case the ZOSHBI sensor is delivered with materials for the user to assemble the RG line.

In case of standard Probostat, the quick connect bulkhead needs to be replaced with a bulkhead. Remove the side plate of the base unit hexagon and remove the Bulkhead quick connect for the Inner In gas line, replace with the supplied bulkhead. The gas line bend is roughly 60° and threading a 1/32" gas line all the way to "inside" the ProboStat can be achieved by utilizing these approaches.

Push the RG line in from the top, grabbing gently with pliers and pushing in about 1 cm at a time, careful not to damage the pins.

The distance of the RG line protruding into ProboStat should be about 5 cm. This length makes it easier to mount the ZOSHBI.

3 External adapter

3.1 Easy solutions

At the outside end of the RG line, the arrangement is flexible. The reference gas used for the oxygen sensor is typically air, and so is the return gas from the sensor. This can be let into the room or led to a vent hood.

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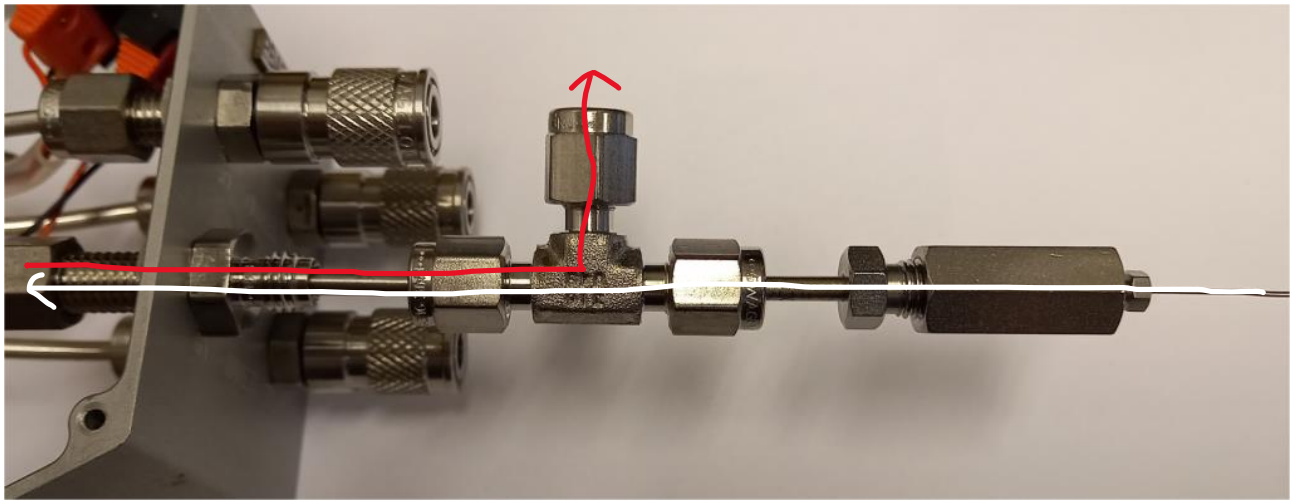
Most convenient and compact arrangement is tiny silicon hose from the 1/32” tube to reference gas source, and return gas connection remains open to room.

Slightly more robust but larger solution is to push the 1/32” through T-joint and glue around the outside of the 1/32” and inside the inside of the T-joint.

Both the above solutions are not as robust as adapters, but they are easier, cheaper and more compact solution for users who wish to dismount and remount the RG line.

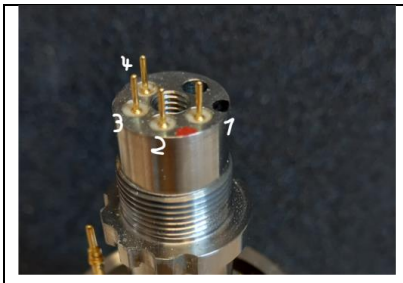
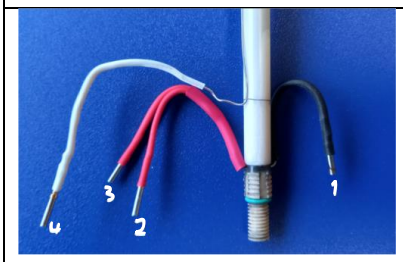
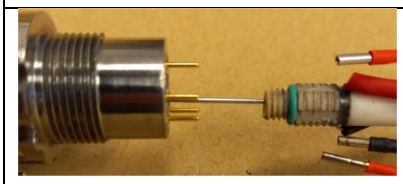
3.2 The adapter

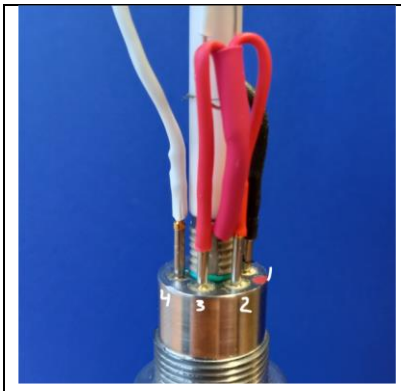
The adapter or materials for it are included with the ZOSHBI setup. The adapter connects to the “Inner In” bulkhead, has a T-joint, then a reducer from 1/8” to 1/32”. The setup also includes suitable size silicone hose to connect and disconnect from the 1/32” gas line.



4 Mounting the sensor

The first step of mounting any measurement setup on ProboStat that utilizes the ZOSHBI sensor, is to mount the sensor itself.

	The ZOSHBI mounts on the Inner In gas line and the four electrode contacts of the inner chamber.
	The sensor has three leads one of which has double purpose. Red and Black form an S-type thermocouple, whereas Red and White measure electrolyte potential. For that reason, the red lead has two ends. The threads on the PEEK adapter may be shorter, this is to avoid excessive rotation when mounting.
	The inside of the oxygen sensor has alumina spacer tube with four holes, only two of which are occupied. It takes a number of tries until the 1/32” gas delivery line enters one of the free holes.
	It may be necessary to add some high temperature vacuum grease on the 1/32” gas delivery line to “seal” it against the inside of the alumina tube hole to discourage the gas from taking a shortcut, and instead take the longer route via the sensor tip.
	Before connecting the electrodes to the pins, it is good idea to check for gas leakages. Block on of the inner in connections and expose the other to slight overpressure, max 0.5 bars. Apply soapy water or soap foam to the bottom of the tube. Tighten the tube until there are no bubbles forming. It may be necessary to take off the O-ring clean that and the stub, and apply new high temperature vacuum grease.

	Mounting the electrode contacts: Pin 1: Sensor Black, Thermocouple + Pin 2: Sensor Red, Thermocouple - Pin 3: Sensor Red, Electrolyte reference side Pin 4: Sensor White, Electrolyte experiment side The nomenclature here is of particular concern, the the immediate visual outside of the sensor tube is also the experiment side, and in that sense the “inside” while what is hidden from the view, the “inside” the sensor tube itself, is flushed with reference gas, and thus the “outside”. To avoid this contradiction, it is best to think of the sides and electrodes from these sides as “experiment side” and “reference side”. The pins are numbered clockwise from the inner out gas.

Bend the three connections aside, ensure the threaded adapter of the sensor has an O-ring. Inside the sensor is an insulation tube with four holes. Two are filled with the electrode lines, and two empty ones. The empty ones can carry the reference gas up and down from the sensor tip.

When mounting the sensor, the first phase is to gentry and carefully move the sensor around until the 1/32” reference gas line enters one of the two free holes. Gently slide the sensor down all the way and screw it to the threads. There is enough space, flexibility and play for the 1/32” gas line to bend to accommodate for the <1 mm radius it rotates along. It is good idea to be gentle and alert for any feedback of potential elevated resistance.

The gap between the inside of the alumina hole and the outside of the 1/32” gas line allows some of the gas to return without going to the tip of the sensor. To prevent this shortcut as potential pathway for the gas, the gap can be filled with high temperature vacuum grease or small piece of thin PTFE tape. Good flow of the reference gas to the tip of the sensor can be verified by altering the flush gas contents or the flow rate. Unfortunately, this validation requires the setup to be heated to several hundred °C, so it is not an easy or quick process.

5 Usage

5.1 Reference gas

Reference gas is sent in from the 1/32” gas line, and taken out from the 1/8” gas line, with a low flow. This directionality ensures there is no pressure buildup inside the ZOSHBI tube.

5.2 Measurements

Both properties to be measured are voltages. The thermocouple voltage between Pin 1 and Pin 2 can be measured from the ProboStat TCBI miniature thermocouple connector. The internal wiring of these contacts inside the ProboStat are appropriate S-type thermocouple compensation wires.

The sensor voltage can be measured between the Pin 3 and Pin 4 designated on ProboStat ILV and ILC accordingly. These are standard BNC panel mounts.

Measurement and calculations can be automated with Omega software (and suitable multimeter). Omega software has functionality for conveniently (measuring and) calculating cold junction compensation, thermocouple voltage to temperature conversion and solving the Nernst equation.

5.3 Thermocouple calculations

Thermocouple voltage to temperature calculations including cold junction compensation are outside the scope of this manual.

5.4 Oxygen partial pressure

In case of oxygen partial pressure measurements with YSZ ceramic the Nernst equation

$$E = \frac{\mathrm{RT}}{4\mathrm{F}} \ln\left(\frac{\mathrm{p}O_2\mathrm{Experiment}}{\mathrm{p}O_2\mathrm{Reference}}\right)$$

Can be conveniently rearranged for calculation of pO_2 in the experiment (formula 1)

$$pO_2\mathrm{Experiment} = \mathrm{p}O_2\mathrm{Reference} \cdot 2.71828^{\left(-46421\frac{E}{T}\right)}$$

Or for verifying operation from known gases (formula 2)

$$E = -0.0000496\ \mathrm{T}\ log_{10}(pO_2\mathrm{Experiment}/\mathrm{p}O_2\mathrm{Reference})$$

Where

E is the sensor electromotive force in Volts

T is the sensor temperature in Kelvins

Or, for conveniency; use E in millivolts, and constants of -46.421 and -0.0496 in the formulas accordingly.

6 Use with Omega

6.1 Measurement

Usage with immediate data feedback is often necessary to get the somewhat complicated process to work. Omega needs to be set to measure sensor voltage, electrolyte voltage, and a temperature used as cold junction compensation. The process is much like using MOSE sensor, except the function used to solve for pO_2 is different.

Say the sensor ZOSHBI thermocouple is connected to TCB/I (pins 1 & 2), the sensor voltage to pins ILC (pin 3) and ILV (Pin 4). If the measurement is done with ProboStat connection box (with DMM6500) these can be connected from ProboStat to TCB and C&D on the connector box. Corresponding channels to measure would be

	ZOSHBI thermocouple	ZOSHBI sensor voltage	Cold junction Temp
Pins on ProboStat	1+(black) & 2-(Red)	3 & 4	-
Connectors on ProboStat	TCB/I	ILC and ILV	-
Connector on connector box	TCB	For example, C & D	-
Channel on switch card	3	9	7 Channel terminates inside the box with a thermistor. It's resistance is accurately a function of temperature inside the box.
Node example name	Node 1 – Zoshbi TC	Node 2 – Zoshbi voltage	Node 3 – Box temp
Node variable prefix	$N1.	$N2.	$N3.
Node type	MV	MV	M2
Node full variable	$N1.MV	$N2.MV	$N3.M2
Instrument commands before measurement (DMM 6500 only)	*CLS :SENS:FUNC "VOLT:DC", (**@3**) :ROUT:DEL 1, (**@3**) :ROUT:CLOSE (**@3**) :READ?	*CLS :SENS:FUNC "VOLT:DC", (**@9**) :ROUT:DEL 1, (**@9**) :ROUT:CLOSE (**@9**) :READ?	*CLS :SENS:FUNC "RES", (**@7**) :ROUT:CLOSE (**@7**) :READ?
Instrument commands after measurement (DMM 6500 only)	:ROUT:OPEN:ALL	:ROUT:OPEN:ALL	:ROUT:OPEN:ALL

6.2 Calculations

With the above setup, in the graphs-section, the NERPO2(E,T,R) expression will calculate/plot the pO_2 of the cell. Firtst however, we need the accurate temperature of the sensor tip: TCS($N2.MV, TT2($N3.M2))

NERPO2($N1.MV, TCS($N2.MV, TT2($N3.M2)), 0.209) where the 0.209 is the reference gas oxygen content in units of ATM.

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7 Build notes & Quality control

In the printed or digital manual, the table below is empty. During production it is printed out, used as reference and filled out.

Zirconia tube length
Disk samples: System length - 11.8 = 38.2 or 48.2 cm
Bar samples: System length - 19.6 = 30.4 or 40.4 cm

Subject	Checked	Notes
Intended system length		50 or 60 cm - Affects tube length
Inner or outer chamber		Default is inner chamber
Intended use		For disc samples or for bar samples - Affects tube length
Resulting length		
Intended adapters		
Check: Thermocouple polarity		
Check: Inside wires bent out of way		
Check: Gas tightness		
Check: Mountable		
Check: Tip at intended location		
Check: Cleaned and packed		
Check: Intended adapters		
Check: O-ring		
Check: Silicone hose		

8 Packing list

- ZOSHBI
- Manuals
- Reference gas line (installed or separate)
- Adapter assembly
- Silicone hose 10 cm