

Miniature Oxygen Sensor Electrode for the ProboStat™

Operating Instruction

Supplementary Material to the ProboStat™ Manual
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Miniature Oxygen Sensor Electrode (MOSE) is combined oxygen sensor and S-type thermocouple mounted ready for use in any standard ProboStat™ configuration. The sensor has a built-in metallic reference: made of a metal and its oxide, it is placed and sealed inside a zirconia sheath. The sensor requires no reference gas and is very small; sensor tip of the electrode is 10 mm long and 3 mm diameter (Fig.1).

By default the MOSE sensor is made to fit 60 cm long ProboStat™ system in the outer chamber, with the center of the sensor tip at the sample height. At special request the MOSE can be manufactured to any length, and can be made to fit also the inner chamber of ProboStat™.

The default model can be used for temperature range from 600°C to 1050°C.

The wires of S-type MOSE have the following colors and functions and recommended connection feedthroughs:

| <i>MOSE wire color</i> | <i>MOSE wire function</i> | <i>Feedthrough code for outer chamber</i> | <i>Feedthrough code for inner chamber</i> |
|------------------------|---------------------------|---|---|
| Red | Thermocouple - / Common | 7 (LVS) | 4 (ILV) |
| White | O ₂ Reference | 8 (LV) | 3 (ILVS) |
| Black | Thermocouple + | 9 (TCT+) | 1 (TCB+) |
| Red | Thermocouple - / Common | 10 (TCT-) | 2 (TCB-) |

The S-type MOSE needs to be used with ProboStat™ that has S-type thermocouple wiring.

Measurements of thermocouple and sensor voltage are made on corresponding connections. Thermocouple voltage can be measured between red and black while sensor voltage is measured between red and white. Sensor voltage must always be measured with voltmeter that has input impedance/resistance of at least 1 GΩ. Lower resistance will allow current to pass through the voltmeter and render the sensor useless.

For the recommended connections MOSE thermocouple voltage would be measured on TCT (feedthroughs 9 and 10), and the sensor voltage - between the LVS and LV (feedthroughs 7 and 8). Utilize a BNC-banana adapter that is shipped with ProboStat™ systems for these measurements in case needed to connect to a measuring instrument.

For the sensor voltage measurements only use a high impedance voltmeter with input impedance over or 1 GΩ. For this same reason we advise against using combinations of shield feedthroughs (5, 7, 14, 16) or LC (6) for carrying sensor voltage. The O₂ sensor voltage electrode must never short circuit with common electrode or connect with the ground potential while the sensor is above room temperature, and using more than one shield pin will allow this possibility (just flick of a switch on ProboStat™ base unit will connect all shield pins).

Other configurations can also be used as long as the thermocouple voltage is carried in the correctly compensated feedthrough.

Principle

Zirconia is a solid electrolyte conducting oxygen ions at high temperatures. An electrochemical voltage appears between the two platinum plated surfaces of the zirconia when they are in contact with two gaseous atmospheres at different oxygen partial pressures. This voltage follows the Nernst equation:

$$E = \frac{RT}{4F} \ln \frac{P_{meas}}{P_{ref}}$$

where

R and F are the constants,

E is the voltage,

T is the temperature,

P_{ref} is the reference oxygen partial pressure,

P_{meas} is the measured oxygen partial pressure.

Knowing the reference oxygen partial pressure, and measuring voltage and temperature, you can calculate the oxygen partial pressure you need to measure.

MOSE, unlike common zirconia sensors called "air reference" sensors, gets its own O₂ reference from a mixture of metal/metal oxide placed and sealed inside it. This avoids using any reference gas.

Calculation of oxygen partial pressure

The calculation of oxygen partial pressure follows the Nernst equation, as below:

$$E = E_0 + \frac{RT}{4F} \ln \frac{pO_2}{0,2095}$$

$$pO_2 = 0,2095 \times 10^{(20,158 \times \frac{E-E_0}{T})}$$

where E_0 is the MOSE voltage (when placed in air).

To calibrate the sensor this measurement is done in several temperatures with known pO_{2ref} so the E_0 can be solved for. The E_0 versus temperature is a linear dependency $y=cx+d$ where line coefficient c is used for calibration factors for the specific sensor.

Final calibration factors are

$B_x = c \times (4 \times 96.487 / 8.315)$ and $A_x = E_0 - (c \times T_0) \times (4 \times 96.487 / 8.315)$. This calibration is done at factory, and the factors A_x and B_x are given with each sensor in addition to an excel sheet.

With these any pO_2 can be calculated using the following formula

$$pO_{2meas} = pO_{2ref} \times \exp\left(\left(46.416 \times \frac{E}{T}\right) - \frac{A_x}{T} - B_x\right)$$

The provided excel sheet, and the factory calibration factors use mV, and % O₂ (1013.25 mbar).

For example when $A_x = 26619.55069$ and $B_x = -25.06457246$:

Sensor voltage of 48 mV at 700°C should result in 0.209 O₂/atm,

Sensor voltage of 20 mV at 700°C should result in 0.055 O₂/atm,

Sensor voltage of -20 mV at 700°C should result in 0.00816 O₂/atm,

Sensor voltage of -200 mV at 700°C should result in 0.00000152 O₂/atm.

Practical operation

Measure the thermocouple voltage with instrument capable of converting it to temperature or with voltmeter and convert yourself.

Measure the sensor voltage with instrument with minimum of 1 G Ω input impedance.

Various versions of the calculator/Excel file exist.

In case more than one sensor was delivered, select correct tab on the accompanying excel file. The name of the tab needs to match the serial number of the sensor. The Excel file is emailed, ask NORECS for it if you do not have the file.

Enter the temperature to the accompanying Excel file at the bottom to cell F48 or F52. Enter the sensor voltage in millivolts to field E48 or E52. Some file versions require user to click calculate to get P1. The Excel file cell addresses given here may vary.

It is also possible to measure the T and pO₂ live with NORECS Omega software and suitable voltmeter.

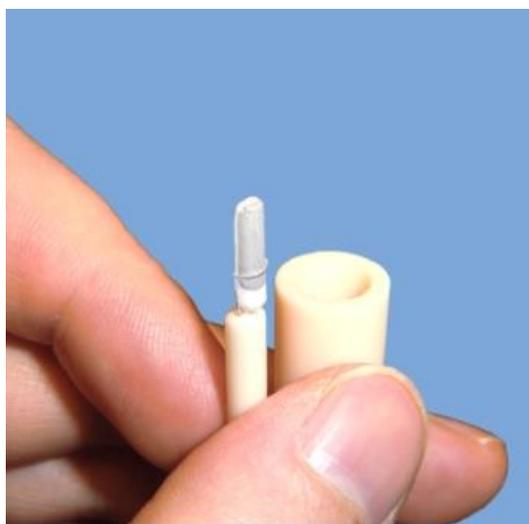


Fig. 1. Tip of the MOSE assembly next to a ProboStat™ sample support tube.

It is very important not to bend the tip of the sensor nor apply any sort of force onto it. The thin Pt and Pt/Rh wires can easily get cut by the sharp alumina tube.

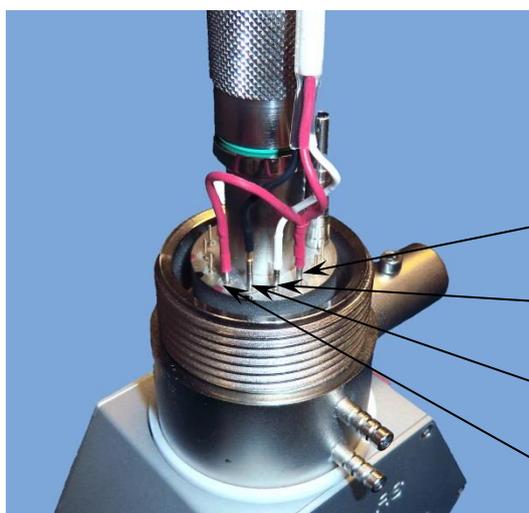


Fig. 2. Suggested connection scheme for S-type MOSE.

Feedthrough 7 (LVS) = Common

Feedthrough 8 (LV) = O₂ sensor reference

Feedthrough 9 (TCT+) = Thermocouple +

Feedthrough 10 (TCT-) = Thermocouple -, Common

Measuring MOSE with Omega software

This chapter is to give the customer idea how it is possible to automate the measurement and calculations with another (not included) product called Omega software. This is not to be taken as exhaustive manual for this subject.

Omega software with suitable multimeter (such as Keithley 2000 with 10 channel switch) can be used to measure all required properties and to calculate the final pO_2 . Instrument will measure two voltages from sensor (and a thermistor resistance) in rapid sequence. From the resistance the software will calculate the necessary cold junction compensation; and use the thermocouple voltage and sensor voltage to solve the Nernst equation for the pO_2 with built-in function for MOSE.

The function to calculate and plot the pO_2 (fraction of pure O_2 at atmospheric pressure) in Omega is

MOSE(T,E,A,B) where

T is the sensor temperature in Celsius,

E is the sensor voltage (with odd results change the polarity),

A is the calibration factor A_x from supplied excel sheet (in cell E54),

B is the calibration factor B_x from supplied excel sheet (in cell E55).

To solve for absolute temperature one can use TCS and TT2 functions explained further in the Omega manual. TT2 converts thermistor resistance to temperature; this is our cold junction compensation. TCS solves for S-type thermocouple temperature.

TT2(R) where

R is the resistance measured on the thermistor. (Located where the thermocouple ends and similar electrical leads go to the measurement instrument)

Thermistor has roughly 10 000 Ω at room temperature, so the above gets evaluated to for example 25°C (actually to our ambient temperature). The thermistor should be located where the thermocouple wires ends, and identical measurement wires start.

The S-type thermocouple conversion function

TSC(V, AM) where

V is thermocouple voltage, and AM is ambient temperature.

First parameter V is the thermocouple voltage from the sensor (black and red legs), second (AM) is the cold junction compensation in Celsius. The cold junction temperature is converted to voltage using the S-type thermocouple conversion function backwards, and added to the given thermocouple voltage V. The sum of voltages is then converted to absolute temperature using S-type thermocouple conversion function.

The final expression in Omega is would look something like **MOSE(TSC(\$N1.MV, TT2(\$N2.M2)), \$N3.MV, A, B)** where \$N1.MV represent thermocouple voltage, \$N2.M2 represents thermistor resistance, \$N3.MV represents sensor voltage, and A and B are the calibration factors typed in by hand by the user. Node 1 (\$N1.) is the channel for sensor thermocouple voltage, Node 2 is resistance of the thermistor located at the end of the thermocouple compensation cables, used as cold junction compensation, and Node 3 is the sensor voltage.