HORIBA

Technical Tip 08

Dissolved Oxygen Electrode Care and Maintenance Procedures

Electrochemical dissolved oxygen (DO) electrodes are composed of anode and cathode, which are submerged in electrolyte solution and enclosed in a cap fitted with hydrophobic, gas permeable membrane. They are also integrated with temperature sensors to measure the temperature of standards and samples and allow the DO meter to compensate the temperature effect on the measured DO values. There are two types of electrochemical DO electrodes: polarographic and galvanic. The advantages of galvanic DO electrodes over polarographic DO electrodes are that they don't require outside voltage source and warm-up time to operate and their electrolyte can be used indefinitely.

Electrochemical DO electrodes available in the market have replaceable electrolyte and either loose membranes or membrane cap assemblies (caps pre-fitted with membranes). The usual maintenance involves periodic changing of the membrane or membrane cap assembly, refilling the electrolyte, and cleaning the anode and cathode. To reduce downtime, the replaceable parts should be readily available and users should know how to perform maintenance.

With the advantages of galvanic DO electrodes and improvement of user experience in mind, HORIBA introduced galvanic DO electrodes equipped with replaceable innovative DO tips for hasslefree plug-and-use operation and built-in temperature sensors for temperature compensation on measured DO values. The DO tips require no maintenance, making them beneficial for both expert and novice users and ideal for continuous measurements in the laboratory or field. Simply, plug the DO tip into the electrode body to perform calibration and measurement and unplug it when the DO electrode will be stored for a long period.

Materials Needed

- Clean water (e.g., distilled, deionized, or tap water)
- Sodium sulfite (Na₂SO₃) or Nitrogen gas
- Cobalt (II) salt (e.g., CoCl₂•6H₂O)
- Soft cotton gauze or tissue
- Pocket barometer

Selection

There are two DO electrode models available, the 9520-10D with 7541 DO tip and 9551-20D (1m cable) / 100D (10m cable) with 5401 DO tip, which are intended for laboratory and field applications, respectively. Electrode bodies are made of durable plastic. The DO tips have lead anode and silver cathode submerged in KOH electrolyte solution and isolated from sample by a gas permeable membrane.

Aside from DO tips, the DO electrodes are also supplied with accessories. The 9551-20D comes with stirrer (and its casing) and an adapter to fit the electrode body into standard biological oxygen demand (BOD) bottles. A magnetic stir plate should be used with the DO electrode for the stirrer to function. The 9551-20D / 100D comes with stainless steel protective guard that shields the DO tip from getting damage and attached cable hook that helps secure the electrode's connection to the meter and prevents the cable from breaking.

DO Electrode Model	9520-10D	9551-20D	9551-100D
Application	For laboratory use	For field use	
DO Range	0 — 19.99 mg/L	0 — 19.99 mg/L	
Temperature Range	0-45 °C	0-40 °C	
Dimensions	184 x 15 mm (1m cable)	165 x 32 mm (2m cable)	165 x 32 mm (10m cable)
Replacement DO tip	7541	5401	

Preparation

Each DO electrode comes with one DO tip. The DO tip is packaged with a drying agent in a sealed aluminum foil bag when shipped. It must be plug into the electrode body before using the DO electrode. Refer to the electrode manual for the procedure.

Calibration

One or two calibration points can be performed on the DO meter / electrode system before measuring samples. For most applications, air calibration is sufficient. Calibration should be checked daily and after relevant changes of ambient conditions (i.e. temperature or pressure). If DO is a critical parameter or sample has low DO value, it is recommended to perform a second calibration point or a check using a zero DO solution.

Set the atmospheric pressure (also called air pressure) in the DO meter before calibrating in either saturation ratio (%) mode, also known as percent saturation mode, or DO (mg/L) mode. If measurement will be carried out at sea level, no need to adjust the default setting 101.3 kPa (equal to 1 atm = 760 mmHg). If measurement is at high altitude, measure the atmospheric pressure with a pocket barometer or check it from your local weather service and enter the value into the DO meter. The atmospheric pressure decreases as altitude increases. Refer to the meter manual for the atmospheric pressure setting and calibration procedures in saturation ratio (%) mode and DO (mg/L) mode.

Air Calibration

This is performed in clean air. There should no water droplets on the membrane or temperature sensor. Evaporation of moisture on the membrane and temperature sensor of DO electrode may influence the readings during calibration.

After air calibration in saturation ratio (%) mode, the meter will show 105%. This is equivalent to 100% saturation in water. HORIBA determined 5% as the difference between the electrode currents in air and in water based on experimental results. In DO (mg/L) mode, the meter will show the actual DO concentration at the measured temperature and set atmospheric pressure. This value divided by the theoretical DO concentration gives the percent saturation.

Zero Calibration

This is performed in a pure nitrogen atmosphere or in an oxygenfree solution. A zero DO solution can be prepared by dissolving 1 g or more of sodium sulfite (Na₂SO₃), an oxygen scavenger, with 1 L of distilled or deionized (DI) water in a container. This solution should be freshly prepared and the volume should be enough to cover the membrane and temperature sensor. If available, 1 mg of cobalt salt (e.g., CoCl₂•6H₂O) can be added in the solution to act as catalyst and indicator. Immediately after preparation, close the container with a cap or film to prevent it from absorbing oxygen and use the solution after an adequate reaction time.

After calibration in saturation ratio (%) mode, the meter will show 0%. In DO (mg/L) mode, the zero DO solution should read less than 0.2 mg/L DO. Rinse the DO electrode thoroughly to avoid contaminating samples.

Slope

After one or two calibration points, the meter will display a slope (%). The slope should be within the range 50 - 200%. If the slope is less than 50% or more than 200%, replace the DO tip.

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Sample Measurement

When measuring samples, take note of the following:

- Measure directly in the water body on site. If not possible, measure discrete sample immediately after sampling.
- The factors that affect DO in samples are temperature, atmospheric pressure, and salinity. DO meters detect the temperature from DO electrodes so make sure that the temperature sensor is submerged in the sample. Adjust the atmospheric pressure and enter the salinity values of samples into the meter, if necessary.
- Stir the sample by using stirrer or moving the electrode to prevent loss of signal due to consumption of oxygen by the DO electrode.
- Avoid formation of any air bubbles in the samples.
- Between measurements, rinse the DO electrode with clean water and blot dry with soft tissue.

Cleaning

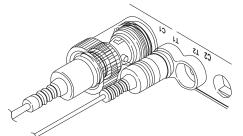
If the membrane is dirty, wipe it gently with soft cotton gauze or tissue then rinse with clean water. Be careful not to scratch the membrane.

Storage

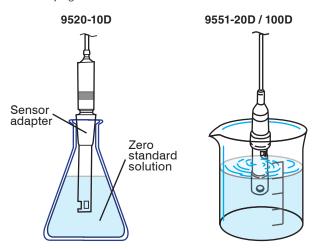
DO electrodes should be clean before they are stored for any length of time.

• Short-term

1. Leave the DO electrode connected to the meter.



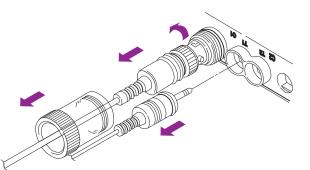
Immerse the DO tip of 9520-10D in zero DO solution (5% Na₂SO₃ solution = 50g Na₂SO₃ in 1L DI water) and the DO tip of 9551-20D / 100D in clean water (e.g., distilled, deionized, or tap water) to prevent the membrane from drying out.



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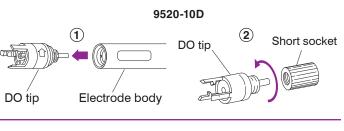
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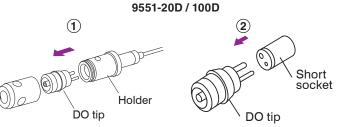


1. Disconnect the DO electrode from the meter.

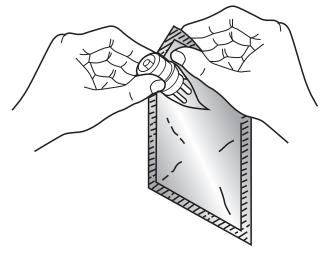
Long-term

2. Detach the DO tip from the electrode and plug the short socket into the DO tip.





3. Wrap the DO tip with aluminum foil. You may reuse the original packaging, if it is still in good condition. Alternatively, place the DO tip in a bottle containing drying agent and store in a cool, dark place.



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