



Vinmetrica Dissolved Oxygen System User Manual

The Vinmetrica dissolved Oxygen system provides a simple, accurate and affordable way to determine oxygen concentrations in wine and other samples.

Materials provided in the kit:

1. Electrolyte Solution (PN: SC-200-13-2)
2. Sodium sulfite powder (PN: SC-200-13-1)
(for 0% Oxygen reference)
3. Vinmetrica Dissolved Oxygen (DO) probe
4. Standard measuring vial
5. One 5 mL syringe (SC-100-6)
6. Extra DO probe membrane (SC-200-13-3)



Figure 1. The Dissolved Oxygen Kit

Things you will need:

1. Vinmetrica SC-200 or SC-300, or comparable meter with 1 mV or 0.02 pH resolution.
2. Distilled water (DI water), which usually can be found at your local grocery store. It's handy to have a wash bottle for rinsing. Rinse bottle available from Vinmetrica (Part number SC-100-17)
3. Small measuring spoon or spatula (e.g., Vinmetrica 0.05 g spoon part#RS-12)

Why Test for Dissolved Oxygen?

Dissolved oxygen (DO) has numerous effects on wine, some desirable, some definitely not. Depending on the type of wine and its characteristics, controlled exposure to oxygen plays a beneficial role in wine quality such as influencing color stability and suppleness. However, too much oxygen exposure can lead to premature deterioration, browning, and acetaldehyde formation. At time of bottling, for example, it is generally desirable to keep DO below 10% saturation. DO can then be measured throughout the bottling process to ensure oxygen uptake is minimized. Careful management of DO will ensure best stability and outcome for your wine.

How it works:

The electrode has an internal electrolyte solution behind a gas-permeable membrane. Oxygen in the sample diffuses into this internal electrolyte and creates an electrochemical (“galvanic”) reaction at the metal contact on the electrode. This is picked up by the SC-200 or -300 and displayed as a voltage reading (firmware x.1.2 or higher), or as an apparent “pH” value, either of which can be related to the response of known oxygen content, allowing you to determine DO as % saturation.

Setup

1. Gently remove the black rubber cover from the end of the electrode.
2. Electrode preparation:
 - a. Remove the end of the electrode (“membrane assembly”) by gently unscrewing it from the barrel. **NOTE: Do not pull off the end cap, this may damage the membrane.** This will expose the silver-gray (zinc) cylindrical anode and its shiny (silver) cathode disk at the end.
 - b. Rinse the inside of the membrane with DI water and shake out excess water thoroughly.
 - c. Hold the membrane assembly in a vertical position and fill to half full with electrolyte. Ensure that no air bubbles are trapped in the electrolyte. Hold the electrode vertically down and screw membrane assembly up until the electrolyte begins to seep up around the threads. Then very slowly continue to screw the membrane module up until it is completely fitted. Then unscrew slowly to release any pressure. Finally, screw membrane fully onto the electrode until it is just tight. **Do not over tighten.**
 - d. Check that the membrane is not pressurized. It should not be bulging out and should be uniformly stretched. Inspect the membrane for tears or leakage. Replace the membrane if any damage has occurred.
3. Attach the electrode to the pH meter and put it in pH mode or, if available, potential mode (Firmware versions x.1.2 or higher, where x is 2 or 3).
 - a. To use potential mode on a SC-200 or 300 turn instrument on and select pH mode, hit Mode button one more time. The pH LED should now be blinking to indicate potential mode.

Procedures

1. Calibration: To calibrate the response of your electrode and instrument, you will determine the values for “0% saturation” (i.e., when no oxygen is present in solution) and for “100% saturation” (i.e., when the maximum amount of oxygen is present).
 - a. Calibrate 0% saturated DO: place about 2 mL of DI water into the measuring vial (about 2/3 full) and add about 0.5 g (less than ¼ teaspoon) of sodium sulfite powder. Swirl for 30 seconds to mix. This is in effect a saturated solution and it removes all the oxygen from the water [note: you can also make a 0% saturated DO solution by bubbling pure nitrogen through DI water at a moderate rate for 5 minutes]. Place the electrode in the vial. Keep the solution moving by gently rotating or swirling the vial. Wait for the value to stabilize, about 2 minutes. In potential mode, you should see a value of 0 ± 10 mV or so. If you are reading apparent pH, you will see a value of around 6.5-7.0, depending on the previous pH calibration settings. Write down the value that you see; this is your **0%DO**.
 - b. Calibrate 100% saturated DO: remove the electrode and rinse it thoroughly with distilled water; gently shake off excess liquid. You can use either method below.

- i. Air method (good, simplest): Allow air to freely contact the membrane. This will make the electrode display an approximate 100% saturated DO value. Typically this is within 5 % of the next method
 - ii. Oxygen-saturated water method (best, more complex): place electrode in distilled water that has been shaken for 3-5 minutes with air, or preferably, that has a continuous stream of air bubbled through it (a cheap aquarium pump works well).
 - c. Wait for the 100% saturated DO value to stabilize, again about 2 minutes. In potential mode, you should see a value of 200 to 300 mV, and always greater than 160. If you are reading apparent pH, you will see a value of around 3 to 2, and always less than 4.5. Write down the value; this is your **100%DO**.
2. Rinse out the measurement vial and shake out excess water. Rinse electrode with DI water.
3. Measure the sample (wine or other liquid) by either method (a.) or (b.) below. Method (a.) avoids contact with your bulk wine or liquid, but is inherently difficult to get highest accuracy because atmospheric oxygen rapidly enters the sample. Method (b.) is easier and accurate, but note the CAUTION statement.
 - a. Take a sample of the wine to be measured. It is important to move quickly to avoid oxygen in the air from reaching the wine sample. (NOTE: if available, use inert gas to sparge the sampling area and the measuring vial to minimize entry of atmospheric oxygen) Place about 2 mL of your wine into the measuring vial (about 2/3 full). Immerse the electrode in the wine sample as in step 1 and slowly swirl the sample. OR
 - b. (Recommended) Insert the clean electrode directly into the wine sample (bottle, carboy, barrel, etc.) and gently swirl. In this case you may want to clean the electrode by dipping it in 20% ethanol solution to minimize contamination of your wine samples. Minimize access of air during this time (sparge the head space with inert gas if possible).
[CAUTION: there is a small risk of affecting your wine by this method. Normally the electrolyte solution will not contact your wine, and vice versa, because the membrane is impermeable except to gases. However in the event of a leakage due to breakage or detachment of the membrane end of the electrode, a small amount of sodium salts and propylene glycol can enter your wine. The amounts are small enough that a bottle or larger size container of wine will not become unsafe to consume, but be aware of the possibility.]
4. Again wait for the reading to stabilize. Usually the millivolt value will drop to a lower reading over about 1 minute, from which it may slowly start to rise again. Note the lowest reading that you see during this time. If you are reading in pH mode, you will see the opposite effect - a low pH reading will rise, then possibly start to decrease again - take the highest value. Table 1 shows some typical data:

Table 1. typical data in mV or pH mode (Note: your data may be different!)

Test	mV	“pH”
0%DO	+3.0	6.87
100%DO	+240	3.48
Wine sample	+36	6.39

5. Calculate DO as % of saturation as follows: Subtract the 0% DO calibration value from each reading. Divide the subtracted value for each wine sample by the subtracted value for the 100% Saturated DO calibration value. Multiply this by 100 to get percent saturated DO for each wine sample, i.e.

$$\text{DO, \% saturation} = 100 * [\text{Wine sample} - \text{0\%DO}] / [\text{100\%DO} - \text{0\%DO}]$$

For example, from the data in Table 1:

$$\begin{aligned} \text{DO, \% saturation} &= 100 * (36-3.0)/(240-3.0) = 14\% \\ \text{Or} &= 100 * (6.39-6.87)/(3.48-6.87) = 14\%. \end{aligned}$$

Reporting dissolved oxygen in mg/L :

If you prefer to get your data in units of mg/L of dissolved oxygen, you can convert your % saturation values. You need to know what value of mg/L DO corresponds to 100% saturation DO. This number depends on several factors, including the conductivity (or “salinity”) of the wine, its alcohol content, temperature, and the ambient barometric pressure. For most wines, only the latter two are significant. At room temperature (72 F or 22.5 degrees C) and ordinary air pressure (sea level on a clear day), the *saturation oxygen level* is about **8.5 mg/L**. For most purposes, this value will be close enough to give you the accuracy needed to make decisions. Be aware, however that this value decreases about 10% for every 5°C (8°F) increase and for every 3000 feet (1000m) of elevation increase.¹

Once you have a value for 100% (saturating) dissolved oxygen for your wine in ambient conditions, calculate your dissolved oxygen from the data you have.

$$\text{DO, mg/L} = \text{saturating oxygen level (mg/L)} \times \% \text{ saturation}$$

In the above example, assuming we are at sea level and 72 degrees, the DO is **8.5** x 14% = 1.2 mg/L

¹ To estimate changes in saturating oxygen level with temperature and pressure, see the dissolved oxygen calculator at <http://water.usgs.gov/software/DOTABLES/>. Set the specific conductivity of the wine to 3000 microsieverts/cm. To calculate air pressure change with altitude, see http://www.altitude.org/air_pressure.php

Finishing Up:

1. Turn off your pH meter and rinse the vials and the electrode with DI water. Replace the black rubber cover over the end of the electrode to prevent damage to the membrane.
2. **Electrode Storage:** The electrode may be stored with its tip submerged in water for a few weeks at a time, but make sure the electrode is disconnected from the meter during storage. For long term storage you should store the electrode dry (unscrew the membrane assembly, drain the electrolyte, rinse everything with distilled water and let dry in air).
3. **Cleaning and Maintenance:** After the electrode has been used for a period of time the residual current may rise, which shows up as an increasing value of 0% DO, and/or you may see a decrease in 100%DO values. First, try replacing the electrolyte as described on page 2 under "Setup". If normal responses still aren't seen within 15 minutes, the electrode may need servicing. To recondition the electrode, gently unscrew the membrane assembly from the electrode body and rinse the inside of the membrane with DI water; allow to air dry. Soak the silver-gray cylindrical zinc anode in 0.1N HCl (1 ml 2M HCl, or the Vinmetrica Acid Solution for SO₂, in 20 ml DI water) for 15 to 20 minutes. Rinse the zinc anode with DI water, blot dry with tissue paper. If necessary, use fine sandpaper or similar fine abrasive to remove any grey or white or other deposits from the anode, and restore a shiny appearance. Rinse with DI water and blot dry with clean cloth or tissue paper. Refill with fresh electrolyte and replace membrane cap as directed in the Setup section. It may take an hour or so for normal responses to stabilize.
4. **Replacing the membrane:** The membrane that comes in the probe has a life expectancy of one year. However the membrane may become damaged and will need replacing. Your kit provides one extra of these membranes (in some cases an additional pre-assembled membrane and cap is already included; in that case there is no need to put a new membrane on it). To replace a membrane, unscrew the electrode cap. You will then pull off the tip of the cap. Remove old or damaged membrane. As gently as possible, remove a new membrane from its paper envelope by gently pulling it off. Place new membrane on top of the larger piece of the cap, then push the tip back into place on top of cap, securing the membrane in place.

Technical assistance: info@vinmetrica.com tel. 760-494-0597

WARRANTIES AND LIABILITIES

1. The materials provided in the kit, as described on page 1 above, (“Materials”) are warranted as follows: All reagents, powders and non-reagent accessories are warranted against defects in workmanship for 6 months from date of purchase. The reagents are warranted to perform as described herein up until any stated expiration date or 6 months after purchase, whichever is later, provided storage recommendations are followed. The electrode is warranted for a period of 1 year. THE WARRANTIES IN THESE TERMS AND CONDITIONS ARE IN LIEU OF ALL OTHER WARRANTIES, EXPRESS OR IMPLIED, INCLUDING WITHOUT LIMITATION ANY WARRANTIES OF MERCHANTABILITY, NONINFRINGEMENT, OR FITNESS FOR A PARTICULAR PURPOSE, SAID WARRANTIES BEING EXPRESSLY DISCLAIMED.
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