Experiment GB-3: Water Quality

Equipment Required

PC or Mac Computer

IXTA, USB cable, power supply

ISE-730 Dissolved oxygen electrode

ISE-100 combination pH electrode

Magnetic stirrer

Stir bars

200 ml flasks (4)

Nalgene plastic water sample bottles with caps

1000 ml beaker

Roll of plastic wrap

pH 4 and pH 7 buffer solutions

Thermometer

Hydrometer and cylinder

Deionized water

Zero-percent oxygen calibration solution or Diet Coke

Dissolved Oxygen Electrode

In 1954, Dr. Leland Clark invented the first membrane-covered electrode designed to measure the concentration of oxygen in blood, solution, and gases. This electrode was innovative because both the anode and the cathode were in a housing that was covered by the same selectively permeable polyethylene membrane. The membrane allowed only a small amount of oxygen to diffuse across it, which reduced the amount of oxygen depleted from the sample. This electrode provided accurate measurements of the oxygen concentration in the sample because the electrons flowing between the cathode and anode were proportional to the concentration of oxygen in the sample

The dissolved oxygen electrode used in this experiment has a Teflon(tm) membrane that permits a limited amount of oxygen to diffuse from the solution being measured to the electrolyte solution inside the removable membrane housing of the electrode. The number of electrons flowing between the cathode and the anode is proportional to the concentration of oxygen that has diffused across the Teflon(tm) membrane into the electrolyte that surrounds the cathode and the anode.

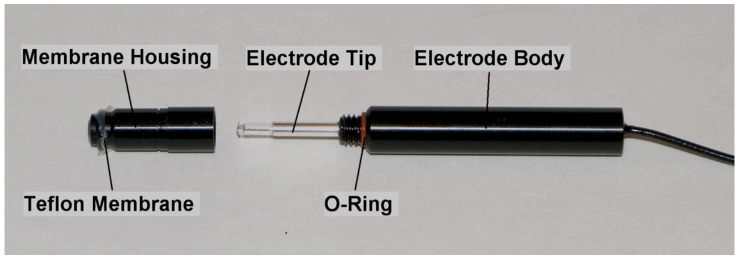

Figure GB-3-S1: The dissolved oxygen electrode, shown with its membrane housing removed. The end of the housing is covered with a Teflon(tm) membrane secured in place by an O-ring. The platinum wire inside the glass tube is the cathode and the silver sleeve that surrounds the glass tube is the anode.

Dissolved Oxygen and pH Electrodes Setup

1. Locate the dissolved oxygen electrode. Plug it into channel A6.
2. Locate the ISE-100 pH electrode and plug it into channel A5.

Warning: When you receive your electrode: 1) Handle it carefully. The tip of the electrode is covered by a delicate Teflon(tm) membrane which can tear easily. 2) Do not tighten or loosen the plastic housing holding the Teflon(tm) membrane. Tightening the housing will stretch or tear the membrane; loosening the housing will cause the electrolyte to leak out of the electrode and affect its responsiveness.

Figure GB-3-S2: The   dissolved oxygen probe and pH probe connected to the IXTA.

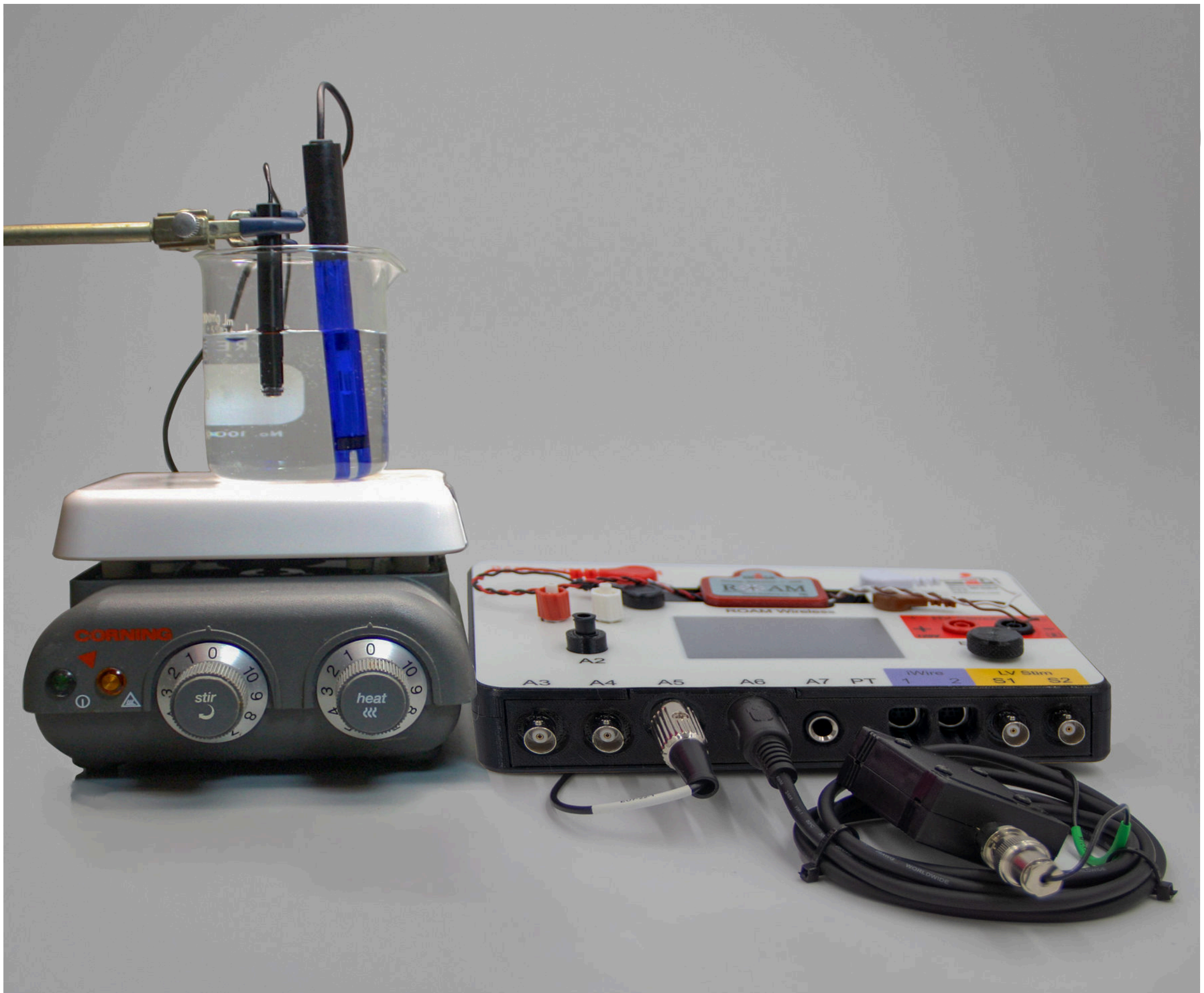

Figure GB-3-S3: The setup for recording oxygen concentration levels and pH using the iWorx/IXTA.

Calibration of the Dissolved Oxygen Electrode

Aim: To calibrate the dissolved oxygen electrode.

The standard used for calibrating the dissolved oxygen electrode is the known concentration of oxygen in air-saturated deionized water. The amount of oxygen that is dissolved in water is known as its solubility (S) and it is dependent upon the temperature, oxygen pressure in the air, and the concentrations of dissolved solutes in the water.

Solubility (S) can be determined by using the following equation:

$$S = (\alpha/22.414)\ ((P-p)/P)\ (r\%/100).$$

In the equation, α is the absorption coefficient of O_2 at the temperature, p is the vapor pressure of water at the temperature, P is the barometric pressure, and r% is the percent oxygen in the air. For example, at 26^oC and 760mmHg and a concentration of oxygen in air of 21%, S equals:

$$(0.02783/22.414L/mole)(734.91mmHg/760mmHg)(0.21) = 252\mu MO_2$$

Procedure

1. Place the oxygen electrode in a 100 ml beaker containing room temperature deionized water. There needs to be enough water in the beaker to submerge the tip of the oxygen electrode, and keep its tip away from the stir bar in the beaker. Place the beaker on a magnetic stirrer. Adjust the speed of the stirrer so the stir bar is rotating quickly and evenly.
2. Type **Saturation-DI Water** in the Mark box.
3. Click Record. The recording will eventually reach a stable level near the top of the recording channel. Click the mark button to mark the recording when the output of the electrode is constant. At this point in the recording, the output of the oxygen electrode is equal to the saturation concentration of oxygen in deionized water at room temperature.
5. Obtain a 100 ml beaker containing zero-percent oxygen calibration solution at room temperature. Make sure there is enough solution in the beaker to keep the tip of the electrode clear of the stir bar.
6. Turn off the magnetic stirrer. Remove the oxygen electrode from the beaker of deionized water and remove the beaker of deionized water from the stirrer.
7. Place the beaker containing zero-percent oxygen calibration solution on the stirrer. Turn on the stirrer and adjust the speed of the stirrer so the stir bar is rotating quickly and evenly. Place the oxygen electrode in the beaker with the zero-percent oxygen calibration solution.
8. Type **No Oxygen** in the Mark box.
9. The recording will eventually reach a stable level near the bottom of the recording channel. Click the mark button to mark the recording when the output of the electrode is constant. At this point in the recording, the output of the oxygen electrode is equal to no oxygen being dissolved in deionized water at room temperature.
10. Click Stop to halt the recording.
11. Select Save As in the File menu, type a name for the file. Click on the Save button to save the data file.
12. Turn off the stirrer. Remove the electrode from the beaker of calibration solution. Hold the electrode over the beaker used for collecting waste liquid, and rinse the electrode with deionized water from a wash bottle. Blot any drops of solution from the electrode and place it in a beaker of deionized water.

Units Conversion-Dissolved Oxygen Probe

1. Measure the temperature (in ^oC) in the lab room. Assume the barometric pressure in the lab room is one atmosphere (760mmHg) and the concentration of oxygen in the air is 21%. From Table 1, find the dissolved oxygen concentration ($[O_2]$) in deionized water at room temperature. This concentration will be used in Step 6 to calibrate the dissolved oxygen electrode.
2. Scroll to the beginning of the calibration data for the dissolved oxygen electrode.
3. Use the Display Time icons on the LabScribe toolbar to adjust the Display Time of the Main window to show the data collected at both the 100% and 0% saturation levels of oxygen in water on the Main window at the same time.
4. Click the Double Cursor icon so that two cursors appear on the Main window. Place one cursor on the flat section of data collected when the saturation of dissolved oxygen in water was 100% and the second cursor on the flat section of data collected when the saturation of dissolved oxygen in water was 0%.
5. To convert the voltages at the positions of the cursors to % Oxygen values, use the Simple Units Conversion dialogue window. To access this dialogue window, click on the arrow to the left of the channel title, O2 Concentration, to open the channel menu. Select Units from the channel menu, and select Simple from the Units submenu.

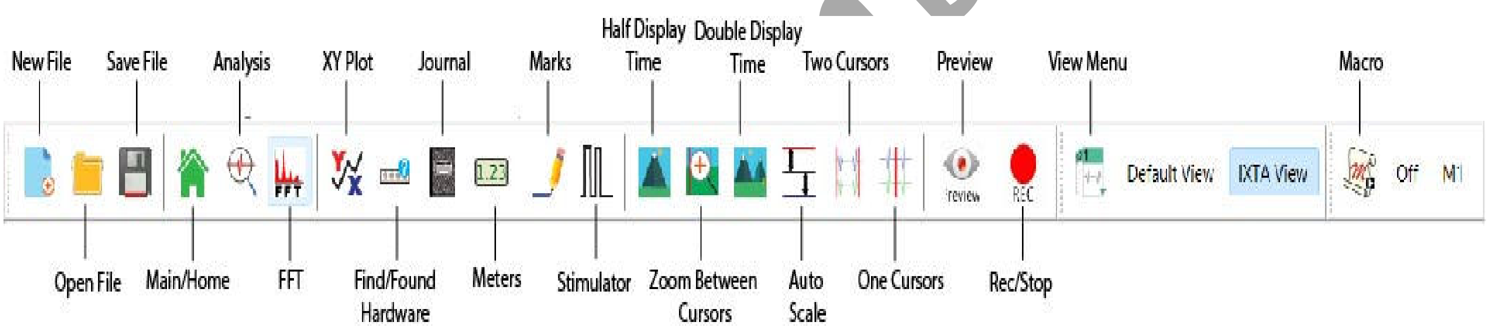

Figure GB-3-S4: The LabScribe toolbar.

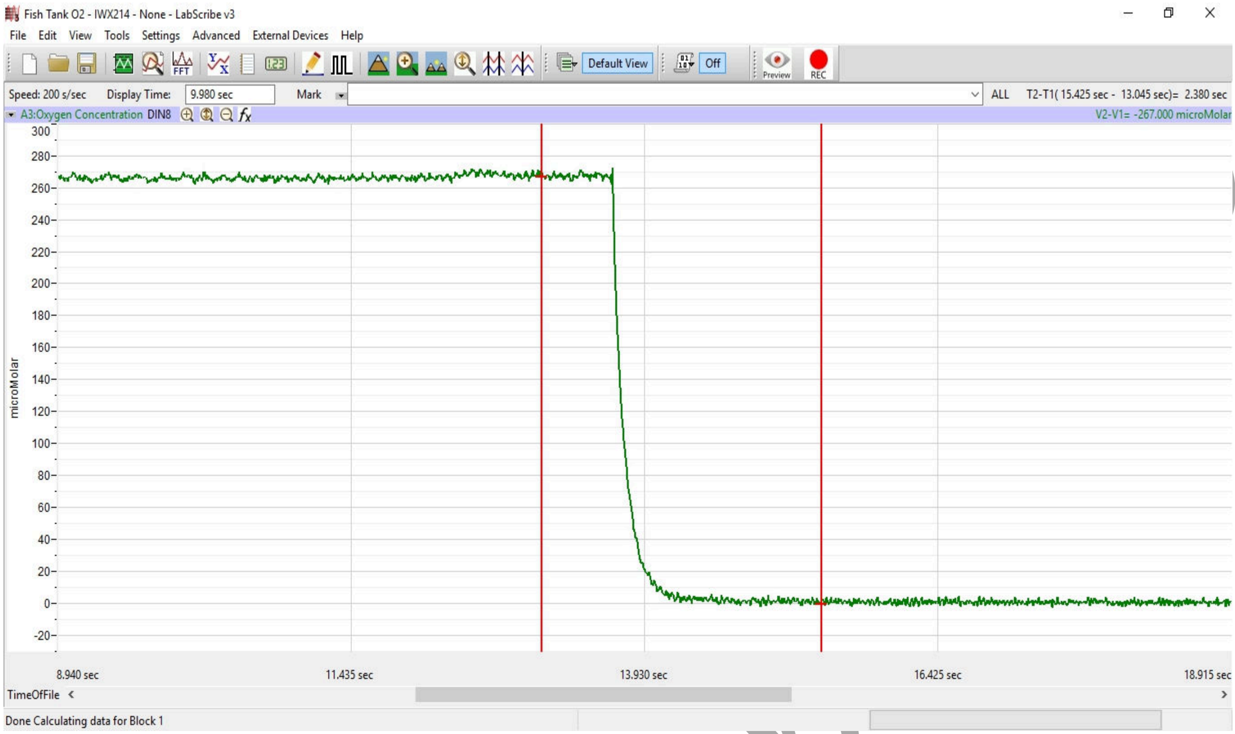

Figure GB-3-S5: Recording of oxygen concentrations in air saturated and oxygen depleted deionized waters used to convert the units of the Y-axis from voltage to O_2 concentration (µMolar).

Table GB-3-S1: Concentration of Oxygen [O2] in Air-Saturated Deionized Water at 1 Atmosphere.

Temp (^oC)	O_2 Abs Coeff (a)	H_2O Vapor Click (p in mmHg)	[O2] (µM)
20	.03102	17.54	284
21	.03044	18.65	278
22	.02988	19.83	273
23	.02934	21.07	267
24	.02881	22.38	262
25	.02831	23.76	257
26	.02783	25.09	252
27	.02736	26.74	247
28	.02691	28.35	243
29	.02649	30.04	238
30	.02608	31.82	234

6. On the units conversion window, make sure 2 point calibration is selected in the pull-down menu in the upper-left corner of the window. Put a check mark in the box next to Apply units to all blocks. Notice that the voltages from the positions of the cursors are automatically entered into the value equations.
7. From the table, find the concentration of dissolved oxygen in water at the room temperature that is 100% saturated. Enter this concentration in the corresponding box to the right of the voltage at 100% oxygen saturation. Enter zero in the corresponding box to the right of the voltage for 0% oxygen saturation.

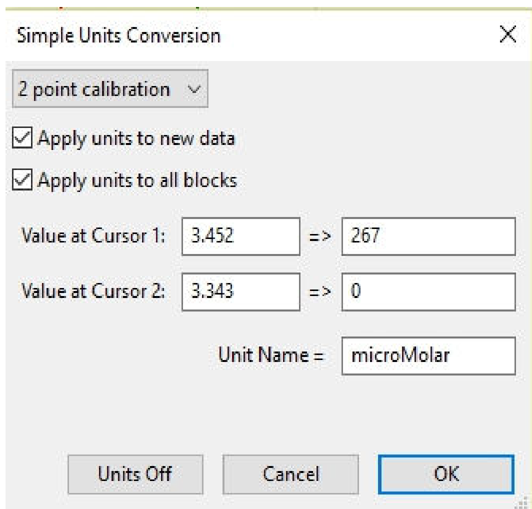

Figure GB-3-S6: The Simple Units Conversion dialogue window with the voltages at the cursors set to equal the dissolved oxygen concentrations used in calibration.

8. Enter the name of the units, µMolar, in box below the concentration. Click on the OK button in the lower right corner of the window to activate the units conversion.
9. Select Save in the File menu, type a name for the file. Click on the Save button to save the data file.

Calibration of the pH Electrode

1. If the ISE-100 pH electrode is still stored in its bottle of buffer, remove the electrode from the bottle. Rinse the electrode with deionized water while holding the electrode over a 1000 ml beaker used for the collection of waste liquids.
2. Place the tip of the ISE-100 pH electrode in a 100 ml beaker containing enough room temperature deionized water to submerge the tip. Keep the electrode in deionized water for at least ten minutes.

3. Prepare two 100 ml beakers, each filled with 50 ml of the pH buffers used for calibrating the pH electrode. The buffers should be at room temperature. Fill one beaker with pH 7 buffer; and the other with pH 4 buffer. Each beaker should be filled with enough buffer to cover the tip of the ISE-100 pH electrode, and also allow the stir bar in the beaker to spin without touching the electrode.
4. Place the beaker containing the pH 7 buffer on the magnetic stirrer. Carefully place a stir bar in the beaker. Remove the electrode from the deionized water and blot any extra drops of water. Position the tip of the electrode in the beaker of pH 7 buffer so that the tip is away from the stir bar. Adjust the speed of the stirrer so the stir bar is rotating evenly at a slow speed.
5. Click Record. After a few seconds, the trace will reach a stable baseline toward the top of the recording channel. Type the words **Calibration - pH 7** in the Mark box. Click the mark button to mark the stable baseline of the recording. This will mark the output of the ISE-100 pH electrode in pH 7 buffer. Continue recording while changing the beakers of buffers.
6. Turn off the stirrer and remove the ISE-100 pH electrode from the beaker of pH 7 buffer. Hold the electrode over the beaker used for collecting waste liquid and rinse it with deionized water. Blot any extra drops of water.
7. Place the beaker of pH 4 buffer on the stirrer. Carefully place a stir bar in the beaker. Position the tip of the electrode in the beaker of pH 4 buffer so that it is away from the stir bar. Adjust the speed of the stirrer so the stir bar is rotating evenly at a slow speed.
8. As you continue to record, the trace will reach a stable baseline toward the bottom of the recording channel. Type the words **Calibration - pH 4** in the Mark box. Click the mark button to mark the stable baseline of the recording. This marks the output of the ISE-100 pH electrode in pH 4 buffer.
9. Click Stop to halt the recording.
10. Click on the Save button to add this information to your data file.
11. Turn off the stirrer. Remove the electrode from the beaker of pH 4 buffer. Hold the electrode over the beaker used for collecting waste liquid, and rinse it with deionized water from a wash bottle. Blot any extra drops of water and place the electrode in a beaker of deionized water.

Units Conversion-pH

1. Scroll to the beginning of the calibration data for the ISE-100 pH electrode.
2. Use the Display Time icons to adjust the Display Time of the Main window to show the data collected at pH 7 and pH 4on the Main window at the same time. The required data can also be selected by:
    - Placing the cursors on either side of data required and,
    - Clicking the Zoom between Cursors button on the LabScribe toolbar to expand the segment of data to the width of the Main window.
3. Click the 2-Cursor icon so that two cursors appear on the Main window. Place one cursor on the flat section of data collected when the ISE-100 pH electrode was in the pH 7 buffer and the second cursor on the flat section of data collected when the electrode was in the pH 4 buffer.

4. To convert the voltages at the positions of the cursors to pH values, use the Simple Units Conversion dialogue window. To access this dialogue window, click on the arrow to the left of the channel title, pH, to open the channel menu. Select Units from the channel menu, and select Simple from the Units submenu.
5. On the units conversion window, make sure 2 point calibration is selected in the pull-down menu in the upper-left corner of the window. Put a check mark in the box next to Apply units to all blocks. Notice that the voltages from the positions of the cursors are automatically entered into the value equations. Enter the two buffers used in the calibration recording in the corresponding boxes on the right side of the conversion equations. Enter the name of the units, pH, in box below the buffer values. Click on the OK button in the lower right corner of the window to activate the units conversion.
6. Select Save in the File menu.
7. Turn off the stirrer. Remove the pH electrode from the beaker of pH 4 buffer. Hold the electrode over the beaker used for collecting waste liquid, and rinse it with deionized water from a wash bottle. Blot any drops of water from the electrode and place it in a beaker of deionized water.

Experiment GB-3: Water Quality

Exercise 1: Dissolved Oxygen Concentration of a Water Sample

Aim: To measure the dissolved oxygen concentration of a water sample.

Approximate Time: 15 minutes

Warning: Use gloves, goggles, and lab coats when handling water samples.

Procedure

1. Place a magnetic stirrer on or next to the base of a ringstand. Open the first water sample bottle and place a stir bar in the bottle. Place the cap on the bottle and turn it until it is tight. Turn on the stirrer and position the stir bar in the center of the bottom of the bottle. Turn off the stirrer before Step 2.
2. Remove the dissolved oxygen electrode from the beaker of deionized water. Blot the drops of deionized water from the electrode. Mount the electrode on the ringstand using a clamp.
3. Remove the cap from the water sample bottle, loosen the clamp holding the dissolved oxygen electrode, and place the tip of the electrode in the water sample as close to the center of the bottle as it will go. Quickly seal the opening of the bottle around the electrode with parafilm or plastic wrap to prevent the exchange of gases between the water sample and the environment.
4. Turn on the stirrer so that the stir bar rotates slowly and evenly. Wait two minutes before recording the dissolved oxygen concentration of the water sample.
5. Click Record. When the recording reaches a stable level on the O2 Concentration channel, type **DO2 and the name of the location** where the sample was taken in the Mark box. Click the mark button to mark the recording.
6. Click Stop to halt the recording.
7. Select Save in the File menu. Complete Step 8 and then proceed directly to Exercise 2.
8. Remove the dissolved oxygen electrode from the water sample. Hold the electrode over the beaker used for collecting waste liquid, and rinse it with deionized water from a wash bottle. Blot any drops of water from the electrode and place it in a beaker of deionized water.

Exercise 2: pH of a Water Sample

Aim: To measure the pH of a water sample which indicates the acidity of the water.

Approximate Time: 15 minutes

Procedure

1. As soon as the dissolved oxygen concentration of the first water sample is recorded, record the pH of the same water sample.

2. Remove the pH electrode from the beaker of deionized water. Blot the drops of deionized water from the electrode. Mount the electrode on the ringstand using a clamp.
3. Loosen the clamp holding the pH electrode, and place the tip of the electrode in the water sample as close to the center of the bottle as it will go.
4. Turn on the stirrer so that the stir bar rotates slowly and evenly. Wait thirty seconds before recording the pH of the water sample.
5. Click Record on the LabScribe Main window to begin recording. When the recording reaches a stable level on the pH channel, type **pH and the name of the location** where the sample was taken in the Mark box. Click the mark button to mark the recording.
6. Click Stop to halt the recording.
7. Select Save in the File menu. Complete Step 8 and then proceed directly to Exercise 3.
8. Remove the pH electrode from the water sample. Hold the electrode over the beaker used for collecting waste liquid, and rinse it with deionized water from a wash bottle. Blot any drops of water from the electrode and place it in a beaker of deionized water.

Exercise 3: Specific Gravity of a Water Sample

Aim: To measure the specific gravity of a water sample which indicates the concentration of dissolved solutes, like sodium and potassium and chloride, in the sample.

Approximate Time: 15 minutes

Procedure

1. Transfer enough of the first water sample to the hydrometer cylinder to fill about 80% of the cylinder.
2. Place the hydrometer in the cylinder and gently spin the device so that it rotates freely for at least two turns.
3. Read the specific gravity of the water sample by matching the meniscus of the sample with the scale on the stem of the hydrometer. The scale ranges from 1.000 at the top to 1.060 at the bottom.
    - If the specific gravity of a water sample is too high (>1.060), measure a given volume of sample and dilute that volume by adding one or two times more deionized water.
    - Account for the dilution when the specific gravity of the sample is recorded. For example, if one volume of deionized water was added to one volume of sample and the hydrometer read 1.02, specific gravity would be calculated by:

        a) Subtracting 1.000 (specific gravity of water) from 1.020 = 0.02;

        b) Multiplying the difference (0.02) x 2 (for 2 volumes) = 0.04; and

        c) Adding 0.04 to 1.000 = 1.04, the specific gravity of sample.
4. Record the specific gravity of the first water sample on Table 1 and in the Journal.

Other Water Samples

Repeat Exercises 1, 2, and 3 for the other water samples that were collected.

Data Analysis

1. Scroll to the section of data recorded during Exercise 1.
2. Click on the 1 cursor icon on the LabScribe toolbar. Place the cursor in the middle of the stable oxygen saturation level recorded on the O2 Concentration channel for the first water sample.
3. Read the dissolved oxygen concentration next to the term Value =, in the upper right margin of the O2 Concentration channel.
4. Record the oxygen saturation level of the first water sample in Table 1 and in the Journal.
5. Scroll to the section of data recorded during Exercise 2. Place the single cursor in the middle of the stable pH level recorded on the pH channel for the first water sample.
6. Read the dissolved oxygen concentration next to the term Value =, in the upper right margin of the pH channel.
7. Record the pH level of the first water sample in the data table and in the Journal.
8. Record the temperature of the first water sample taken at the collection site to the data table and the Journal.
9. Repeat Steps 1 through 8 for each of the other water samples.

Questions

1. Which sample had the highest dissolved oxygen concentration? What could be the causes for that sample to have that level of oxygen saturation?
2. Which sample had the lowest dissolved oxygen concentration? What could be the causes for that sample to have that level of oxygen saturation?
3. Which sample had the highest pH level? What could be the causes for that sample to have that pH level?
4. Which sample had the lowest pH level? What could be the causes for that sample to have that pH level?
5. Which sample had the highest specific gravity? What could be the causes for that sample to have that level of specific gravity?
6. Which sample had the lowest specific gravity? What could be the causes for that sample to have that level of specific gravity?
7. Does your data indicate any correlation between pH level and oxygen saturation level, or oxygen saturation level and specific gravity, or any other combination of parameters?

Table GB-3-3: Values for Four Parameters Recorded from Water Samples

Water Sample/Location	Temperature °Celsius	Dissolved Oxygen Concentration (µMolar O_2)	pH	Specific Gravity
1				
2				
3				
4				
5				
6				
7				