STUDENT STUDY PROJECT

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Title of the project: **ESTIMATION OF DISSOLVED OXYGEN**

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ESTIMATION OF DISSOLVED OXYGEN

Introduction

The Winkler Method is a technique used to measure dissolved oxygen in freshwater systems. Dissolved oxygen is used as an indicator of the health of a water body, where higher dissolved oxygen concentrations are correlated with high productivity and little pollution. This test is performed on-site, as delays between sample collections and testing may result in an alteration in oxygen content.

The Winkler Method uses titration to determine dissolved oxygen in the water sample. A sample bottle is filled completely with water (no air is left to skew the results). The dissolved oxygen in the sample is then "fixed" by adding a series of reagents that form an acid compound that is then titrated with a neutralizing compound that results in a color change. The point of color change is called the "endpoint," which coincides with the dissolved oxygen concentration in the sample. Dissolved oxygen analysis is best done in the field, as the sample will be less altered by atmospheric equilibration.

Principle of Analysis

The chemical determination of oxygen concentrations in seawater is based on the method first proposed by Winkler (1888) and modified by Strickland and Parsons (1968). The basis of the method is that the oxygen in the seawater sample is made to oxidize iodine ion to iodine quantitatively; the amount of iodine generated is determined by titration with a standard thiosulfate solution. The endpoint is determined either by the absorption of ultraviolet light by the tri-iodide ion in the automated method, or using a starch indicator as a visual indicator in the manual method. The amount of oxygen can then be computed from the titer: one mole of O_2 reacts with four moles of thiosulfate.

More specifically, dissolved oxygen is chemically bound to Mn(II)OH in a strongly alkaline medium which results in a brown precipitate, manganic hydroxide ($MnO(OH)_2$).

After complete fixation of oxygen and precipitation of the mixed manganese (II) and (III) hydroxides, the sample is acidified to a pH between 2.5 and 1.0. This causes the precipitated hydroxides to dissolve, liberating the Mn(III) ions. The Mn(III) ions oxidize previously added iodide ions to iodine. Iodine forms a complex with surplus iodide ions. The complex formation is desirable because of its low vapor pressure, yet it decomposes rapidly when iodine is removed from the system. The iodine is then titrated with thiosulfate; iodine is reduced to iodide and the thiosulfate is oxidized to tetrathionate. The stoichiometric equations for the reaction described above are:

$Mn^{2+} + 2OH^{-} \rightarrow Mn(OH)_2$

$2Mn(OH)_2 + 1/2\ O_2 + H_2O \rightarrow 2MnO(OH)2$

$2Mn(OH)_3 + 2I^{-} + 6H^{+} \rightarrow 2Mn^{2+} + I_2 + 6H_2\ 0$

$I_2 + I^{-} \leftrightarrow I_3^{-}$

$I_3^{-} + 2S_2O_3^{2-} \rightarrow 3I^{-} + S_4O_6^{2-}$

The thiosulfate can change its composition and therefore must be standardized with a primary standard, typically potassium iodate. Standardization is based on the co-proportionation reaction of iodide with iodate, thereby forming iodine. As described above, the iodine binds with excess iodide, and the complex is titrated with thiosulfate. One mole of iodate produces three moles iodine, and amount consumed by six moles of thiosulfate.

$IO_3^{-} + 8I^{-} + 6H^{+} \rightarrow 3I_3^{-} + 3H_2O$

$I_3^{-} + 2S_2O_3^{2-} \rightarrow 3I^{-} + S_4O_6^{2-}$

Applications

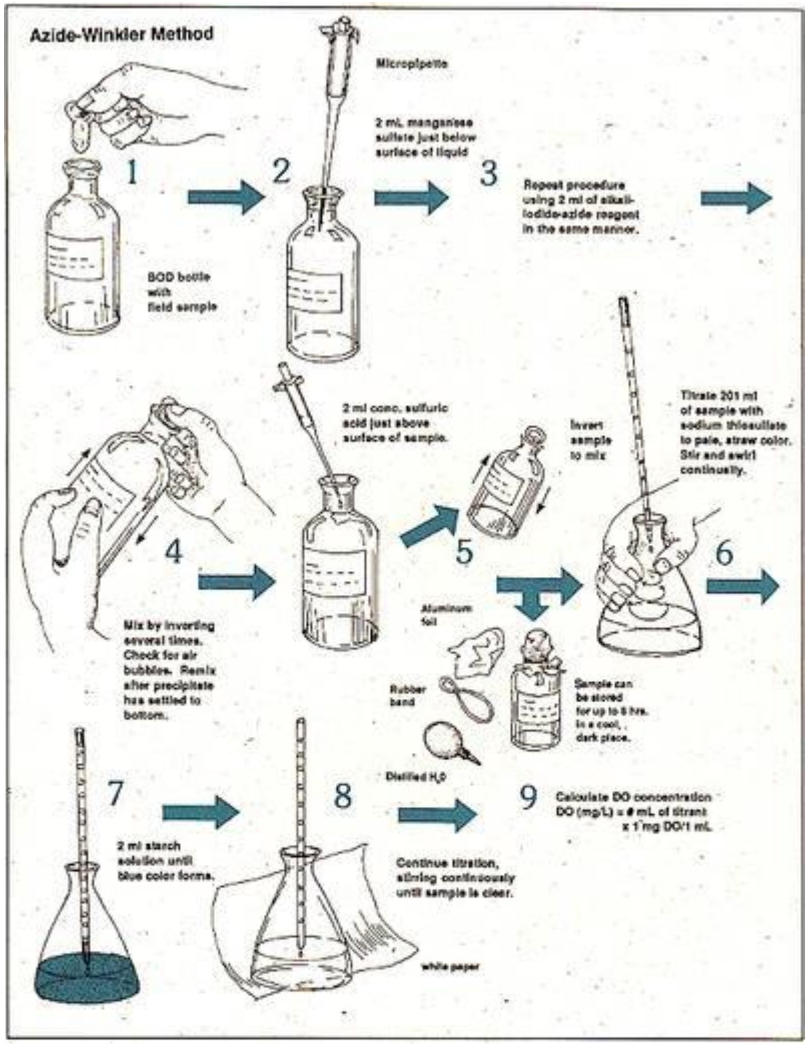
Dissolved oxygen analysis can be used to determine:

the health or cleanliness of a lake or stream,

the amount and type of biomass a freshwater system can support,

the amount of decomposition occurring in the lake or stream.

How to- Sample Collection, Preparation, Analytical Protocols, and Concerns



Dissolved oxygen should be measured as quickly and carefully as possible. Ideally, samples should be measured in the field immediately after collection.

Reagent List:

2ml Manganese sulfate

2ml alkali-iodide-azide

2ml concentrated sulfuric acid

2ml starch solution

Sodium thiosulfate

These reagents are available in dissolved oxygen field kits, such as those made by the Hach Company. Please use caution when using these reagents, as they can be hazardous to one's health.

Procedure:

Carefully fill a 300-mL glass Biological Oxygen Demand (BOD) stoppered bottle brim-full with sample water.

Immediately add 2mL of manganese sulfate to the collection bottle by inserting the calibrated pipette just below the surface of the liquid. (If the reagent is added above the sample surface, you will introduce oxygen into the sample.) Squeeze the pipette slowly so no bubbles are introduced via the pipette.

Add 2 mL of alkali-iodide-azide reagent in the same manner.

Stopper the bottle with care to be sure no air is introduced. Mix the sample by inverting several times. Check for air bubbles; discard the sample and start over if any are seen. If oxygen is present, a brownish-orange cloud of precipitate or floc will appear. When this floc has settle to the bottom, mix the sample by turning it upside down several times and let it settle again.

Add 2 mL of concentrated sulfuric acid via a pipette held just above the surface of the sample. Carefully stopper and invert several times to dissolve the floc. At this point, the sample is "fixed" and can be stored for up to 8 hours if kept in a cool, dark place. As an added precaution, squirt distilled water along the stopper, and cap the bottle with aluminum foil and a rubber band during the storage period.

In a glass flask, titrate 201 mL of the sample with sodium thiosulfate to a pale straw color. Titrate by slowly dropping titrant solution from a calibrated pipette into the flask and continually stirring or swirling the sample water.

Add 2 mL of starch solution so a blue color forms.

Continue slowly titrating until the sample turns clear. As this experiment reaches the endpoint, it will take only one drop of the titrant to eliminate the blue color. Be especially careful that each drop is fully mixed into the sample before adding the next. It is sometimes helpful to hold the flask up to a white sheet of paper to check for absence of the blue color.

The concentration of dissolved oxygen in the sample is equivalent to the number of milliliters of titrant used. Each mL of sodium thiosulfate added in steps 6 and 8 equals 1 mg/L dissolved oxygen.

Results Analysis

The total number of milliliters of titrant used in steps 6-8 equals the total dissolved oxygen in the sample in mg/L. Oxygen saturation is temperature dependent - gas is more soluble in cold waters, hence cold waters generally have higher dissolved oxygen concentrations. Dissolved oxygen also depends on salinity and elevation, or partial pressure. Click here to access an online oxygen saturation calculator that takes these parameters into account. Otherwise, use the chart below to find saturation at a given temperature.

Temperature-Oxygen Solubility Relationship	
Temperature (ºC)	Oxygen Solubility (mg/L)
0	14.6
5	12.8
10	11.3
15	10.2
20	9.2
25	8.6
100	0

Conclusion:

In conclusion, an increase in temperature and an increase in light increase the amount of dissolved oxygen within a water sample. Seeing that the results were consistent through multiple experiments, it is highly likely that if the experiment was repeated that similar or the same results would occur. There were two pieces of data that did not follow the rest of the data collected. It would be interesting to retest these two examples more accurately to see if the information changed or remained the same. This data can help different business and agencies with their environmental goals. Because dissolved oxygen levels impact the organisms living in an environment, agencies and organizations can use the data to predict how these levels affect the organisms under different conditions. Ex: If it is winter, according to our data, an agency can predict that the number of organisms would drop because there is less dissolved oxygen in the water