

WATER QUALITY TESTING BASICS FOR WORK IN THE ENVIRONMENTAL FIELD

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INTRODUCTION

Many geologists have careers in the environmental field. The Bureau of Labor Statistics predicts a 15% growth rate for careers in the environmental industry over the next decade, therefore there will not be a shortage of job opportunities for those looking to apply their background in geology to improve the natural environment for human use and consumption. The environmental sector is a diverse field, both in terms of the professionals who work in it and what they do. Professionals with backgrounds in geology, chemistry, biology, physics, or engineering will work to prevent, control, and remediate environmental problems that relate to surface water, groundwater, soil, air, and waste materials. Depending on the position, a professional may be responsible for quality testing, assessment, remediation, treatment, and/or management of these natural resources and human related waste materials. Most entry level positions will require field and/or laboratory work including collection and sample management and handling, sample testing for any number of analytical parameters, and then data analysis and assessment. This knowledge and skill set goes beyond basic chemistry classes and typically can be gained in an aqueous geochemistry course or water quality course.

The objective of this short course is to provide participants with either an initial exposure to or a refresher of basic water and wastewater quality tests that are commonly used in the environmental industry. This is a hands-on short course that will be run in the new Environmental Technology laboratory at SUNY Canton. Participants will be instructed on how to properly test for common water and wastewater quality parameters such as pH, temperature, conductivity, turbidity, dissolved oxygen, alkalinity, hardness, inorganics, and biochemical oxygen demand (BOD). The test procedures and equipment are those commonly used in environmental field sampling, drinking water treatment plants, and wastewater treatment plants. Using surface/ground water samples collected from around the region participants will learn how to calibrate and use the equipment, execute the analytical test procedures, and interpret the results. Participants will also learn about proper sample collection, handling, and preservation techniques. A variety of equipment/approaches will be used in the analytical tests: probes, meters, titrations, and color spectrophotometers. All methods and procedures are after industry standard and practice found in the ASTM Book of Standards, Volume 11 for Water and Environmental Technology (ASTM International) and Standard Methods for the Examination of Water and Wastewater (APHA, AWWA, and WEF); and in accordance with equipment specific standard operating procedures.

BASIC LAB SAFETY

Safety is of utmost importance and is not something to be taken lightly. Some of the equipment and materials that are used in water and wastewater testing can be potentially very harmful. It is therefore important to know how to properly use and handle these materials and equipment in the field/laboratory setting. Creating and maintaining a safe and healthy environment is a shared responsibility of everyone. This is something that must be taken seriously by all participants. Industry requires training and certifications to address such issues (e.g. OSHA 10hr safety training, OSHA 40 Hour HAZWOPER). The following subsections review key laboratory safety procedures that participants will follow in today's short course. It is not intended to be a fully comprehensive laboratory safety training course.

Protective Equipment

- *Lab Coat:*

Lab coats will be required. They act as a barrier to protect skin and clothing from potentially harmful chemicals, dyes, or other substances encountered in the lab. Be aware of sleeves as they can knock equipment over. Lab coats must be removed if leaving the lab room, so as to not contaminate areas outside of the work space.

- *Safety Glasses or Goggles:*

Eye protection is required when working in the lab. Prescription eyeglasses may be worn instead of safety glasses, provided that they adequately shield the eyes. Some safety glasses will fit over prescription eyeglasses. This is recommended if adequate protection cannot be offered from regular glasses alone. It is strongly encouraged that contact lenses not be worn in the lab. If something is splashed into the eye, it is more difficult to rinse the eye properly with a lens in the way. Trying to remove the lens could cause a delay in rinsing the eye and result in serious damage. Some chemicals will adhere a contact lens directly to the eyeball making it next to impossible to remove.

- *Protective Gloves:*

Gloves should be worn when working with chemicals and potentially infectious materials. Gloves should be replaced if they develop a tear or perforation. When gloves are removed, take them off while turning them inside out and do not touch the exterior of the glove. Properly dispose of the used gloves in a trash can, do not set them down on a counter, table, or workbooks as this may contaminate the surface. If you must leave the lab area, gloves must be removed so as to not contaminate areas outside of the work space.

- *Clothing:*

Proper clothing should be worn at all times. Loose fitting clothing, especially loose shirt sleeves, should be avoided. Long sleeve shirts and pants will provide more protection for your skin than T-shirts and shorts.

- *Footwear:*

Proper footwear should be worn at all times. In particular, no sandals, flip-flops, slippers, ect. should be worn, since they provide no protection should equipment/chemicals be dropped on them.

Chemicals

- *Labeling and Safety Information:*

All chemicals and solutions should be properly labeled so it is clear what they contain. The phone number and web address to obtain the Material Safety Data Sheets (MSDs) are posted in the work area.

- *Handling and Transport:*

Chemical/solution containers should be kept well away from the edge of the workbench and any equipment that generates a flame or excessive heat. When working with chemicals on the workspace the containers should be covered and sealed whenever not in use to prevent spillage. It should not be necessary to transport any chemicals during the short course. However, if chemicals must be moved it should be done carefully, in a fashion that minimizes the chance of a spill or leak. No chemicals should be moved without the permission and supervision of the overseeing faculty member or laboratory assistant. Other participants should be made aware that a chemical is "on the move". While being moved the container should be properly sealed, should possibly be contained in a secondary containment vessel, and the person moving the chemical should be wearing all necessary protective covering.

- *Disposal:*

Not all wastes (chemicals, solutions, containers, disposable equipment, ect) can be disposed of in a normal trash can or down the sink – special secondary containment or pre-disposal treatment may be required. Do not dispose of any wastes down the sink or in the trashcan until you have consulted with the short course instructor.

Equipment

- *Handling of equipment:*

Treat all equipment with care and respect. Most of this equipment is expensive and to varying degrees – fragile. Use equipment only for what it is intended for, do not move equipment unless you are told to do so, and follow the instructors use and handling of the equipment.

- *Cleaning:*

Keep a clean workspace. Each group and individual is responsible for cleaning the equipment and workstation space as instructed. Glassware typically requires a multi-step cleaning – soap wash, 3x tap water rinse, and 3x distilled water rinse. Be careful when handling wet glassware as it becomes very slippery and can easily be dropped into the sink. Should glassware break the instructor will clean and dispose of the broken pieces.

Emergency Response

At the start of the short course the instructor will point out the location of the following:

- Fire extinguishers
- Eye wash fountains
- Emergency showers
- First aid supplies
- MSDS sheet contact information
- Laboratory exits

Miscellaneous Rules

- Follow the instructions provided to you by the instructor at all times. If you are unsure of part of the procedure do not hesitate to ask for assistance.
- *Food and Drink:* Absolutely NO food or drink is allowed in the laboratory area. All food and drink should be consumed prior to coming to the laboratory session. Water is available down the hall at water fountains.
- *Jewelry:* Rings, watches, and bracelets may catch on equipment. Some articles will react negatively with protective gloves and certain chemicals, causing discoloration or staining. In the event that there is a chemical spill, the material may become trapped under the jewelry, making it difficult to remove the chemical and prevent injury. Use precaution when wearing these items in the laboratory.
- *Hair:* Long hair should be kept tied back, especially when working with open flame or when leaning over the bench to work as it may knock equipment or get into chemicals.
- *Hand Washing:* Hands should be washed and dried thoroughly with soap prior to the start and at the end of the lab. Antibacterial soap can be found at each of the work stations.

BASIC WATER QUALITY PARAMETERS

Introduction and Background:

Environmental assessment typically will include measurement of basic water quality parameters such as pH, temperature, conductivity, dissolved oxygen, and turbidity. These parameters are important as they drive most physical, chemical, and/or biological reactions that impact water and wastewater quality and are important factors in treatment/remedial systems. In this section of the short course, participants will learn about the probes and meters used to measure these parameters, how to handle and calibrate the equipment, and how to use the instrumentation to obtain analytical values for pH, temperature, conductivity, dissolved oxygen, and turbidity. Participants will be divided into four (4) groups and each group will be assigned a different source of water. Analytical results from each group will be shared with all participants to allow for comparison and discussion of the results. The same work groups and water sample will be used for the other three short course modules as well.

In water, hydrogen ions, H^+ , are directly or indirectly involved in numerous chemical reactions; and are often driving factors in these reactions. The concentration of hydrogen ions is measured as pH, or acidity, where pH is the negative log concentration of hydrogen ions:

$$pH = -\log [H^+]$$

A solution that is acidic will have a low pH (thus a high concentration of hydrogen ions) and a solution that is basic will have a high pH (a low concentration of hydrogen ions). In order to measure pH a meter and probe are used. The pH scale ranges from values of 0 to 14, with a value of 7 being neutral, pH values less than 7 are considered acidic, and pH values greater than 7 are considered basic. As reference, vinegar has a pH of 2.5, milk has a pH of 6.7, and Pepto Bismol has a pH of about 10. The pH of natural waters (surface and groundwater) can be quite variable depending on overall water quality, bedrock/soil types, seasonal variation, the types of chemical reactions that are occurring, etc. Commonly pH levels of streams in the North Country (e.g. the Grasse River and Raquette River) are close to neutral (6.8-7.2). Municipal drinking water treatment plants have a target pH of about 7.0 in their effluent stream. In water and wastewater treatment systems, some chemical and/or biological reactions require a particular pH in order to proceed or be most efficient (time it takes for a particular treatment process and/or degree to which a reaction will occur). For example, the alum coagulation/flocculation process in a conventional drinking water treatment plant requires a pH of 6. In passive treatment systems for iron acid mine drainage a high pH (8-10) is required to optimize iron oxidation.

Temperature is a factor in both natural and engineered systems as it is one factor that impacts the rate in which many reactions proceed. Both chemical and biological reaction rates typically increase as temperature increases. Water samples are collected and put on ice in the field and stored in a cold refrigerator ($< 4^{\circ}C$) in order to slow both chemical and biological reactions. A temperature probe is typically part of the pH meter and probe.

Conductivity measures the solutions ability to conduct an electronic charge. It is a reflection of the number of ions present in the sample. These ions are charged particles and the electron activity that they produce is a reflection of the ions concentration. The greater the number of ions present in the sample, the greater the conductivity. A probe is used to measure conductivity.

Oxygen is found in water and is referred to as dissolved oxygen (DO). It is typically consumed in many chemical and biological reactions and is an important factor in 1) assessing the water quality for ecological systems, 2) system components of wastewater treatment systems, and 3) assessing water quality of treated

wastewater streams (as participants will learn in the biochemical oxygen demand module of this short course). Dissolved oxygen is measured with a DO meter and probe. A healthy, natural stream would typically have DO concentrations of 7-9 mg/L.

Turbidity is the measure of clarity of water and is used in treatment design tests (e.g. jar test to determine chemical coagulant dose in drinking water treatment) and for monitoring effectiveness of treatment systems. Turbidity is measured with an instrument called a turbidimeter, which measures the amount of light that is able to pass through a sample. The more material that is present in a sample (e.g. solids, metals, bacteria, organics), the less light is able to pass through the sample, and the higher the turbidity of the sample. Turbidity is reported in units of NTU or nephelometric turbidity units. Finished treated drinking water should have a turbidity of 0.1 to 1.0 NTU according to the US Environmental Protection Agency (EPA).

Objectives:

- *Overview of Field Work:* review sample collection, preservation, and handling techniques
- *Analytical tests:* learn how to operate meters and probes used to measure pH, temperature, conductivity, DO, and turbidity, conduct analytical tests for these basic water quality parameters (pH: ASTM D1293-99R05, APHA 4500-H+; temperature: ASTM D6764, APHA 2550; conductivity ASTM D1125-95RR99, APHA 2510; dissolved oxygen ASTM D0888-03, APHA 4500-O; turbidity ASTM D1889-00, APHA 2130), and discuss results from varying water sources

Overview of Field Work:

In the environmental engineering profession, quality control is of the utmost importance. Reducing error and producing repeatable data sets is essential to proper analysis and evaluation. It is therefore necessary to learn the appropriate method in which to collect a water sample so that you can be assured that your data represents the conditions of the water/wastewater source as accurately as possible. The main goal is to collect a number of samples that are a good representation of the water/wastewater source. To ensure that a representative sample is obtained, one must minimize the sampling bias that could arise during collection by having a routine that deals with sample site selection, sample handling, sample preservation, the frequency of collection, and the equipment and method(s) used to collect the sample. Prior to sampling it needs to be determined what parameters are going to be tested. It should then be determined by what method they will be analyzed. The standard procedure for that method should be carefully reviewed. In the standard procedures for that method it should indicate how the sample should be collected: type of sample container, sample volume, sample preservation, and allowable sampling holding time and conditions. Any or all of these may vary depending on the source type (e.g. stream, lake, groundwater well, tap/pipe). All of this should be carefully reviewed to ensure that all of the proper equipment has been acquired and all procedural steps are accurately followed.

As an introduction to measurement of basic water quality parameters, the instructor will give a brief overview of the topics outlined below that relate to field work:

- *Typical field kit:* cooler, ice, clip board, chain of custody forms, sample labels, field book, sample bottles, filtration equipment, preservation materials, samplers (sample bottle, sample cup on arm, bailer, peristaltic pump+tubing), field meters and probes (e.g. pH probe, DO probe, turbidimeter), calibration standards and reagents, distilled/deionized water, gloves, safety glasses, markers, hip boots, water level tape, measuring tape, camera, site map.
- *Sample collection:* containers and collection method
- *Sample preservation:* filtration, chemicals, cooling agent

- *Sample handling*: coolers, chain of custody, arrival in lab, holding times (**Exhibit 1** – Example Chain of Custody form)

Materials for Analytical Tests:

The following materials will be used to determine pH, temperature, conductivity, DO, and turbidity of the water sample.

- pH/temperature probe
- Multiparameter field probe (can test for pH, temperature, conductivity, total dissolved solids, and salinity)
- pH standard solutions
- Conductivity standard solutions
- DO probe
- DO calibration sleeve
- HACH turbidimeter
- HACH turbidity calibration standards and verification sample
- Bottle of distilled water
- 250 mL beakers
- Kim wipes
- Protective gloves
- Water samples

Procedures for Analytical Tests

The following procedures will be used to determine pH, temperature, conductivity, DO, and turbidity of the water sample. Record the results in the data table found in **Exhibit 2**. These results will be shared with the group for discussion of variation in water quality between the different sources.

- Measuring pH, Temperature, and Conductivity
 1. Turn on the pH/temperature/conductivity multimeter probe.
 2. Temperature does not require any calibration.
 3. To calibrate the probe for pH make sure it is reading pH by pressing the “Mode/Ent” button
 4. Rinse the probe with distilled water into a rinse beaker.
 5. Obtain the container with 7.00 standard solution and pour a small amount into the probe cap.
 6. Place the probe into the 7.00 standard solution.
 7. Hit “Cal”.
 8. Wait until the reading is stable and then hit “Mode/Ent”.
 9. Remove the probe from the 7.00 standard solution.
 10. Rinse the probe with distilled water into the rinse beaker.
 11. Dump the calibration standard from the cap into the rinse beaker and repeatedly rinse the cap with distilled water.
 12. Repeat steps 3-9 with the 4.01 and 10.00 pH standard solutions.
 13. To calibrate for conductivity press “Mode/Ent” until the probe is in the conductivity mode.
 14. Rinse the probe with distilled water into a rinse beaker.
 15. Obtain the container with 84 μ S standard solution and pour a small amount into the probe cap.
 16. Place the probe into the 84 μ S standard solution.
 17. Hit “Cal”.
 18. Wait until the reading is stable and then hit “Mode/Ent”.
 19. Remove the probe from the 84 μ S standard solution.
 20. Rinse the probe with distilled water into the rinse beaker.

21. Dump the calibration standard from the cap into the rinse beaker and repeatedly rinse the cap with distilled water.
22. Repeat steps 15-21 with the 1413 μS and 12.88 mS conductivity standard solutions.
23. The probe is now calibrated.
24. Pour approximately 100mL of water sample into an empty 250mL glass beaker.
25. Rinse with probe with distilled water into the rinse container.
26. Place the probe into sample, holding it off the bottom of the beaker and away from the sides of the container.
27. Gently stir the sample with the probe.
28. Monitor the parameters on the screen of the probe, allowing them to equilibrate before taking a reading. Record your results in the table found in **Exhibit 2**. Pressing the “Mode” button will cycle the meter through the different parameters.
29. Remove the probe from the water, rinse with deionized water into the rinse container, gently dry the exterior of the probe with a Kim wipe, replace the end-cap, and turn off the probe.

- Measuring DO

1. Prior to the start of the lab, the DO meter was turned on to allow the probe to polarize. If the meter has automatically shut itself off turn it on.
2. The water-saturated air method is used to calibrate the meter and probe.
3. Take the white calibration sleeve, remove the cap from the one, and take out the gray sponge.
4. Saturate the sponge with distilled water, squeeze out the excess water, replace the sponge into the end of the calibration sleeve, and replace the cap.
5. Make sure there are not water drops at the end of the DO probe – if any are present, gently blot dry with a Kim wipe.
6. Insert the DO probe into the calibration sleeve.
7. Select calibrate (“Cal”) on the DO meter. The meter will cycle through a few readings, should obtain a reading close to 102.3%, and then go back into measurement mode, reading DO at mg/L or %. Press the “Mode” button until the meter reads DO in units of mg/L.
8. To measure DO of the sample, remove the DO probe from the calibration sleeve and suspend it in the beaker that contains the water sample. Holding the beaker in one hand and the probe in the other, gently stir the sample with the probe to get the water sample flowing past the membrane at the end of the probe. Press “Measure” and continue to slowly stir the sample until the DO reading stabilizes. Record the result in the data table found in **Exhibit 2**.

- Measuring Turbidity

1. Turn on the turbidimeter with the On/Off button that looks like a circle with a vertical line through the top.
2. Push the calibration key, which looks like a two point scatter plot with a best fit line.
3. Follow the instructions on the display. Three standard solutions will be used to calibrate the meter. The first calibration standard has a turbidity of 20 NTU, the second of 100 NTU, and the third of 800 NTU. For each calibration standard, carefully remove it from its storage place, hold it at the top being sure not to touch the sides of the vials. Carefully wipe the sides with a Kim wipe to remove any marks. Gently invert each standard several times to stir up the “sediment” that is creating the turbidity for the sample. Do not shake as it may introduce air bubbles that will cause error in the calibration. Insert the vials into the holding cell with the arrow head facing to the front, close the lid, and press “Read”. The display will show the meter stabilizing, will present the result, and will then ask for the next standard. Repeat these steps for each calibration standard.
4. After the 3rd calibration standard has stabilized, press “Done” and then “Store” to save the results.

5. The meter will then require a verification sample to be read. Gently wipe and invert the verification sample cell, place it into the meter's hold cell, close the lid, and press "Read". If the verification sample reads correctly the meter is calibrated and it will automatically re-enter the measurement mode.
6. Once the machine is calibrated test the water sample provided to the group.
7. Obtain the water sample. Gently invert the sample container to mix, but not vigorously as you don't want to add air bubbles as this may impact the results.
8. Rinse the turbidity sample cell two or three times with a small portion of sample.
9. Pour the sample into the sample cell up to the fill line and put on the cap.
10. Using a KimWipe, wipe the entire cell free of fingerprints, water drops, or other marks. Holding the cell up to a source of light is useful for this.
11. Gently invert the cell a couple of times to mix the contents. Again, do not shake as tiny air bubbles can alter readings.
12. Place the sample cell into the turbidimeter with the arrow head facing front, close the lid, and press "READ".
13. Record the turbidity value (units are NTU) in the provided data table found in **Exhibit 2**.

ALKALINITY AND HARDNESS TITRATIONS

Introduction and Background

Alkalinity is defined as the ability of water to neutralize acids. This is commonly referred to as buffering capacity and works to prevent a drop in pH. Alkalinity is the sum of all bases found in water that are titratable with a strong acid. It is essentially the opposite of acidity, which can be defined as the presence of a weak acid preventing a rise in pH upon the addition of a strong base. Typically, alkalinity in surface waters is derived from carbonates, bicarbonates, and hydroxides. These carbon sources may or may not be naturally occurring. If they are naturally occurring, it is usually due to the presence of a certain rock type (e.g. limestone, dolomite, etc.). When one makes the assumption that only the inorganic carbon is significantly impacting alkalinity, it can be defined as follows in equivalents/liter (eq/L):

$$\text{Alkalinity} = [\text{HCO}_3^-] + [\text{CO}_3^{2-}] + [\text{OH}^-]$$

Anions such as borates (e.g. $\text{B}_4\text{O}_5(\text{OH})_4^{2-}$), phosphates (e.g. PO_4^{3-} , HPO_4^{2-} , H_2PO_4^-) and silicates (e.g. $\text{SiO}(\text{OH})_3^-$) may also affect the alkalinity of water. If concentrations of these ions are significant, they must be included in the above equation. Water bodies with high alkalinity (acid neutralizing capacity) are capable of maintaining a pH near neutral pH during environmental events such as acid rain, spring runoff, and acidic or caustic chemical spills. Water with low alkalinity tends to be corrosive, which can be bad for pipes in distribution systems or structures made of reinforced concrete or steel.

While alkalinity could be determined by measuring the ions that are contributing to it, the most common method to determine alkalinity is a titration called the Gran Method. According to the Gran Method, alkalinity can be calculated using the following equation:

$$\text{Alkalinity} = 5000 * (V_e * N_t) / V_s = \# \text{ mg/L as CaCO}_3$$

Where V_e = volume of titrant at the equivalence point (L), N_t = normality of titrant in equivalents per liter (eq/L), V_s = sample volume (L), and the 5000 converts from eq of CaCO_3 to mg of CaCO_3 . The normality of the titrant and the volume of sample are predetermined, so the only value that needs to be determined is the volume of titrant at the equivalence point.

The equivalence point is the point in the titration where an equivalent or stoichiometric amount of titrant has been added to the sample water to complete the acid-base reactions that are occurring, thus depleting the sample of all of the alkalinity. There are several ways to determine the volume of titrant at the equivalence point. The simplest method to determine V_e , is to add an indicator dye to the water sample. An indicator dye, such as Bromphenol blue indicator solution, will be added to the water sample at the beginning of the titration. As the titration proceeds, the equivalence point will be reached when there is a persistent color change. The volume of titrant used at the point of this color change is the V_e needed to calculate alkalinity. See **Table 1** below for an example of a data set recorded from an alkalinity test.

Table 1. Example data set from an alkalinity test.

Example Alkalinity Test Data		
Vs (L) =	0.2	
Nt (eq/L) =	0.1	
Volume of Acid Added (mL)	pH	Gran Function (F1)
0.5	7.33	9.37809E-09
1.0	7.19	1.29777E-08
1.5	7.00	2.015E-08
2.0	6.91	2.48514E-08
2.5	6.69	4.13452E-08
3.0	6.52	6.1305E-08
3.5	6.41	7.91707E-08
4.0	6.15	1.44421E-07
4.5	5.76	3.5538E-07
Color Change	5.0	3.98
	5.1	3.81
	5.2	3.91001E-05
	5.3	5.52572E-05
	5.4	6.64661E-05
	5.5	7.46125E-05
	5.6	8.57083E-05
	5.7	9.84542E-05
	5.8	0.000113096
	5.9	0.000121243

The second way to determine the volume of titrant at the equivalence point is to add incremental volumes of titrant, recording the pH change with each addition, and create a titration curve from this data set (cumulative volume of acid added vs pH). If a strong acid is added to a sample with alkalinity and the sample is not complex (i.e. having many species contributing to the alkalinity), a graph similar to **Figure 1** can be generated. As more and more acid is added, the alkalinity buffers the water sample from initially dropping quickly in pH. However, around the equivalence point there is a rapid change in pH where the moles of weak base converted equals the moles of strong acid added and all of the alkalinity is exhausted. The point where there is a change

in curvature from convex to concave, or visa versa, is termed the equivalence point. The volume of titrant used to reach this point is the V_e needed in the alkalinity equation.

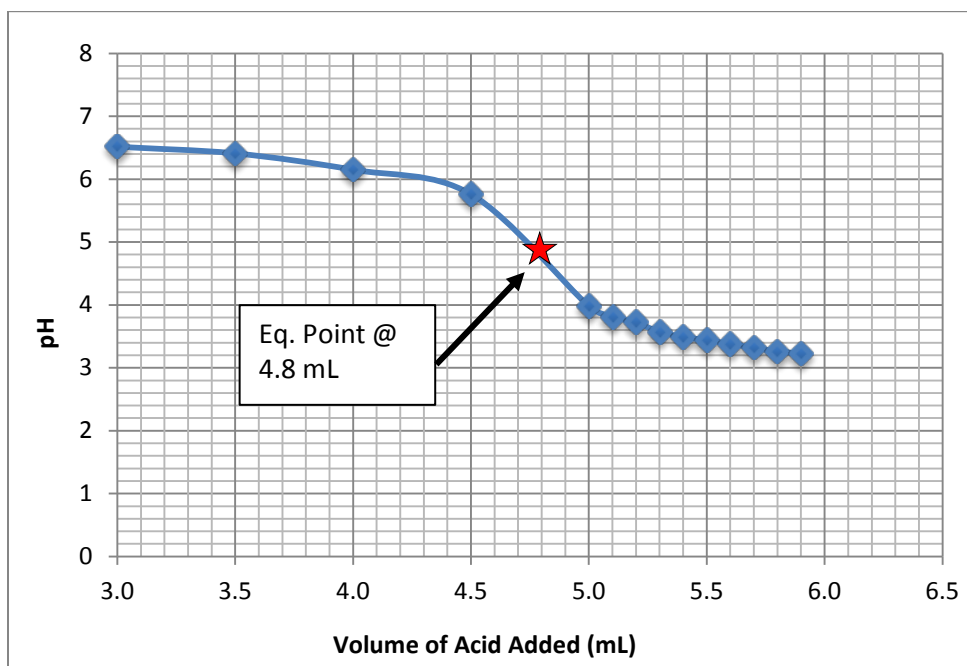


Figure 1: Example alkalinity titration curve.

There are a number of reasons why developing a titration curve can become difficult. One difficulty lies with the type of acid and based found in the water being titrated. When dealing with monoprotic acids and bases (accepts one H^+), the titration curve will look like the one drawn in **Figure 1**. However, if the acid or base accepts more than one hydrogen ion, the curve will become more complex, with multiple equivalence points, creating “steps” in the titration curve. In complex water samples where there are multiple influences on the alkalinity, or if the system is buffered, the equivalence point can become difficult to find or non-existent. In addition, if the alkalinity is very low, it may be difficult to locate the equivalence point on the titration curve. There are multiple ways in which these difficulties can be overcome to determine the volume of titrant used to reach the equivalence point in order to solve for alkalinity. One method requires calculating what is referred to as the first Gran function (F_1). This function is calculated with the following equation:

$$F_1 = (V_s + V_t) * 10^{(-pH)}$$

The first Gran function is determined for each volume of added titrant. Then a plot of F_1 vs volume of added titrant is created. **Figure 2** below is an example of a Gran plot. Note how there is a significant change in slope just as the data approaches a F_1 value of zero. The linear regression line is only plotted through the points on the portion of the data that has a high slope. The linear regression line will intercept the x-axis at the volume of titrant used to reach the equivalence point (V_e). An arrow is used in the plot below to show where the linear regression line intersects the x-axis. This value is the volume at the equivalence point (V_e).

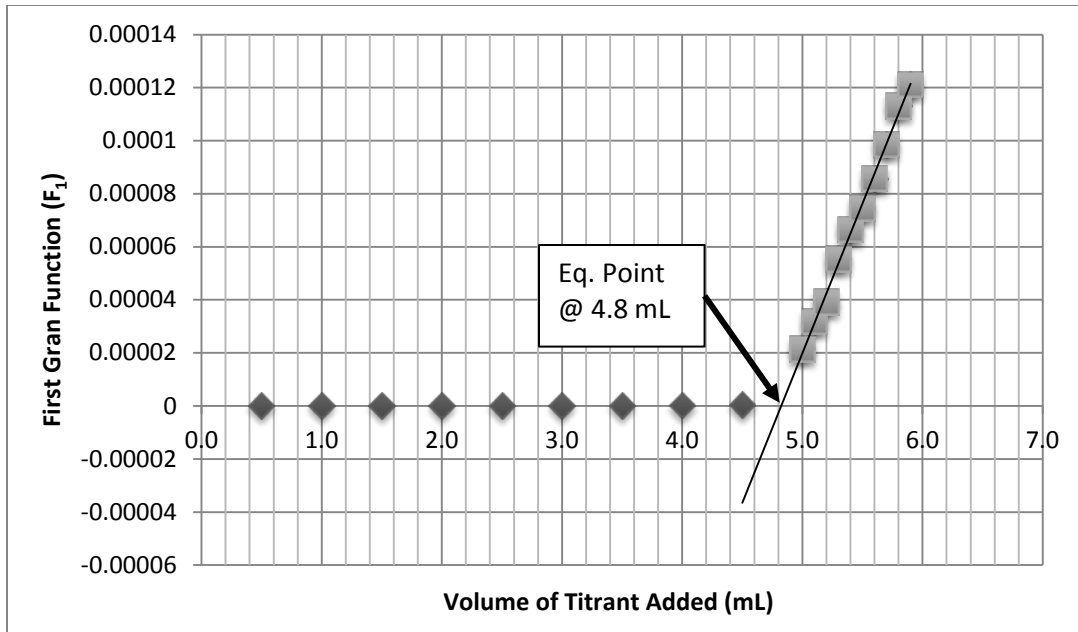


Figure 2. Example of a first Gran function plot for alkalinity.

For natural waters, the equivalence point usually occurs around a pH of 4.5, but this will depend greatly on the geology of the region. It is worth saying that larger volumes of titrant can be added at pH values higher than 5.5, but after this point, smaller volumes should be titrated so as not to miss the equivalence point. Once the volume of titrant at the equivalence point has been determined, it and the sample volume and titrant concentration can be used to calculate alkalinity. Alkalinity is a very important water quality parameter in several remedial/treatment processes. Many treatment processes require controlling/driving certain chemical reactions by manipulating pH and alkalinity. For example, passive treatment systems for iron acid mine drainage contamination requires using limestone channels before and between oxidation and settling ponds in order to increase alkalinity, increase pH, and as a result oxidize ferrous and ferric iron into settleable iron oxide compounds.

Hardness was originally associated with the capacity of water to precipitate soap. The polyvalent metal ions associated with water hardness (chiefly Ca^{2+} and Mg^{2+}) can cause the precipitation and buildup of carbonate or the formation of soap scum. In current practice, the hardness of water is defined as the sum of the magnesium and calcium concentrations and is expressed as mg/L of CaCO_3 . When the hardness is greater than the sum of the carbonate and bicarbonate alkalinity, that amount of hardness equivalent to the total alkalinity is called the carbonate hardness. Hardness in excess of this is called non-carbonate hardness. Water is considered soft when the measured hardness is less than 60 mg/L as CaCO_3 and is considered very hard when greater than 180 mg/L as CaCO_3 . The hardness of the water can be impacted by many factors, soil and/or bedrock type being two of the more significant contributing factors. Groundwater typically is hard whereas surface water bodies tend to be softer. This is why most residences with private wells have water softener systems installed as the general consumer tends to prefer soft water as is reduced carbonate scaling, can prevent soap scum, and can produce a good lather with soap. However, excessively soft water can be corrosive to pipes.

The preferred method of hardness determination is through the separate quantification of calcium and magnesium concentrations where:

$$\text{Hardness (mg/L as CaCO}_3\text{)} = 2.497 (\text{Ca in mg/L}) + 4.118 (\text{Mg in mg/L})$$

This method is more accurate in determining a hardness concentration, but requires two separate analytical techniques. If the results do not warrant this level of accuracy, a single titrimetric technique can be applied that determines the calcium and magnesium hardness simultaneously. The theory behind this technique is outlined briefly below.

Ethylenediaminetetraacetic acid and its sodium salts (EDTA) are added to the solution where they form a complex compound that bonds to metal cations, essentially removing them from the solution as free ions. The amount of EDTA that must be added to remove all of the Ca^{2+} and Mg^{2+} directly correlates to the concentration of these cations – regardless of valence it is always a 1:1 reaction of EDTA:cations. EDTA is titrated into the sample which contains an indicator dye (either Eriochrome Black T or Calmagite). The endpoint of the titration is identified where there is a color change in the solution, indicating that all of the Ca^{2+} and Mg^{2+} have been removed.

The sharpness of the titration endpoint improves with increasing pH. To prevent CaCO_3 and/or $\text{Mg}(\text{OH})_2$ precipitation, the pH for this method is set at a maximum of 10.0 with the stipulation that the titration be completed within 5 minutes. In addition, Mg^{2+} ions must be present in solution to provide a sharp endpoint. To accomplish this and eliminate the need for Mg^{2+} correction, a small amount of EDTA-Mg complex is added to the buffer solution (although this step can prove problematic in very soft waters where this small volume of EDTA can induce an immediate color-change). The EDTA hardness is determined using the following equation:

$$\text{Hardness (EDTA) as CaCO}_3/\text{L} = \{A * B * 1000\} / \{\text{mL sample used}\}$$

Where A = volume of titrant used in the titration and B = mg CaCO_3 equivalent to 1.00 mL of titrant.

The variable B depends on the concentration of the titrant: the higher the concentration, the higher the value of B. This value is determined by titrating a standard concentration (1.000 g/L) of reagent-grade CaCO_3 with the titrant to be used in the laboratory. This value is often provided on the EDTA solution bottle.

Objectives

- Learn how to perform a titration.
- Quantify the alkalinity (ASTM D1067-02, APHA 2320B) and hardness (ASTM D1126-02, APHA 2340) of a water sample and discuss results from different water sources.

Materials for Measuring Alkalinity

The following materials will be used to determine alkalinity of the water sample.

- Mixing plate and magnet
- Stand with burette clamp
- Burette
- pH probe
- 500 mL beaker
- Graduated cylinder
- DI water
- Bromphenol Blue Indicator solution
- 3 mL dropper pipette
- 0.1N Hydrochloric Acid (HCl) titrant
- Safety gloves
- Safety goggles
- Lab coats

Procedure for Measuring Alkalinity

The following procedure will be used to determine alkalinity of the water sample. Record the pH and titrant volumes in the alkalinity data table found in **Exhibit 3**. The final calculated alkalinity value can be recorded in the data table found in **Exhibit 2**. The final alkalinity value will be shared with the group for discussion of variation in water quality between the different sources.

1. Obtain a graduate cylinder and measure 200 mL of sample water.
2. Transfer sample into a 500 mL beaker.
3. Place a magnetic stirrer into the beaker, place on stir plate under burette found in the fume hood, turn on stirrer. The setup should be positioned beneath the burette.
4. Add 7+ drops of Bromphenol blue indicator solution (note that more may be required to add a noticeable blue color to the solution – check with the instructor).
5. Rinse pH probe with deionized water from squirt bottle and place into sample, submerging the end, but not allowing it to touch the bottom/sides of the beaker.
6. Record the initial volume of acid titrant and initial pH.
7. Slowly add acid titrant in small volumes (0.5 mL), unless pH change is greater than 0.2 units, then adjust accordingly. With each addition of titrant, record the cumulative volume of titrant added and the new pH value.
8. At a pH of about 5.5, decrease the volume of titrant, adding only 0.1 mL or as little as a few drops at a time. With each addition of titrant, record the new cumulative volume of titrant added and the pH.
9. When color change remains constant (blue changes to yellow), record the titrant volume and pH – you have reached the equivalence point.
10. Continue to add titrant in 0.1 mL increments, recording the volume and pH, until you've reached a pH of about 3.
11. Calculate V_e (in L) based on the Bromphenol Blue Indicator dye color change:

$$V_e = (\text{titrant volume at color change}) - (\text{initial titrant volume})$$

12. Calculate the alkalinity (in mg/L CaCO_3):

$$\text{Alkalinity} = 5000 * (V_e * N_t) / V_s$$

Where V_e = volume of titrant at the equivalence point (L), N_t = normality of titrant in equivalents per liter (eq/L), V_s = sample volume (L), and 5000 converts from eq to mg.

13. Should time permit the groups may collectively work with the instructor to calculate alkalinity using the graphical methods.

Materials for Hardness

The following materials will be used to determine hardness of the water sample.

- Stand with burette clamp
- Burette
- Mixing plate
- Magnet

- 250 mL beaker
- Graduated cylinder
- DI water
- Eriochrome Black T Indicator solution
- Buffer Solution
- EDTA titrant
- 3 mL dropper pipettes
- Safety gloves
- Safety goggles
- Lab coats

Procedure for Hardness

The following procedure will be used to determine hardness of the water sample. Record the titrant volumes in the hardness data table found in **Exhibit 4**. The final calculated hardness value can be recorded in the data table found in **Exhibit 2**. The final hardness value will be shared with the group for discussion of variation in water quality between the different sources.

1. Use a graduated cylinder to measure the water samples. Obtain 100 mL of sample water and place it in a 250 mL beaker.
2. Place the beaker on the mixing plate below the burette found in the fume hood.
3. Place a magnet bar in the beaker and turn on the mixing plate.
4. Add 4 to 8 drops of indicator solution (either Eriochrome Black T) to the sample (note that more may be required to add noticeable color to the solution – check with the instructor).
5. Add 2 mL of the buffer solution. Note – the titration must be completed within 5 minutes of buffer addition. This is to minimize the tendency of the CaCO_3 to precipitate from solution. Should the addition of the buffer solution result in a color change – get a new sample volume, follow steps 1 through 4, and skip step 5 (do not add buffer solution).
6. Record the initial volume of EDTA by reading the volume on the burette.
7. Add EDTA titrant slowly (a rate of 1-2 drops per second is ideal), until the last pink tinge disappears. Add the last few drops at 3 to 5 second intervals. At the end point, the solution is normally blue. The indicator dye changes from pink to blue over the span of one drop so care is required. If the endpoint color change is missed the titration will have to be repeated.
8. Record the final volume of EDTA titrant.
9. Calculate the volume of EDTA titrant used.
10. Calculate the hardness (in mg/L CaCO_3).

INORGANIC ANALYSIS

Introduction and Background

Metals (inorganic ions, such as iron, manganese, aluminum, arsenic, uranium, and lead) are found in varying concentrations in natural waters. Typically these metals are released from bedrock via dissolution from contact with water. Initially these metals will be present in their dissolved, ion state. In groundwater the metals typically will remain in a dissolved state as groundwater typically has low oxygen concentrations and so oxidation reactions do not occur. In surface water these metals tend to oxidize to some extent, due to high oxygen, microbial oxidation, and reactions with organic material. Presence and concentrations of these

minerals can be quite variable depending on rock type, degree of exposure to water, and water quality. In areas where there has been industrial mining for metal-bearing minerals (e.g. pyrite releases iron), surface water runoff over mine tailings piles can often result in concentrations higher than typical background levels due to natural exposure. Some of these metals may also make their way into waterways from other industrial processes wastewater discharges.

The presence of these metals may or may not be a water quality issue depending on the metal type and concentration. When considering metal concentrations for drinking water purposes, metals like iron, manganese, and aluminum are regulated for aesthetic purposes, not health related concerns. The secondary maximum contaminant levels (SMCL) for these metals is not enforced by the EPA, but in areas where these compounds are found at higher levels drinking water treatment plants will treat for these metals and will meet the SMCL. Metals like arsenic, uranium, and lead do cause health problems, have enforceable maximum contaminant levels (MCLs), and must be treated for in drinking water treatment plants. Metals analysis can be routine in water quality assessment, remediation/treatment design, and remedial/treatment quality control testing. Therefore, it is important to have an understanding of how these materials should be properly sampled and analyzed.

In this module participants will analyze water samples for iron. As mentioned above, iron is a nuisance compound that leads to problems such as red water, staining of laundry and food, pipe fouling and interference of other technical equipment. The drinking water standard for iron is 0.30 mg/L. Iron can be present as ferrous iron (Fe^{2+}), ferric iron (Fe^{3+}), or any number of iron compounds (e.g. $\text{Fe}(\text{OH})_3$ or Iron(III) hydroxide). Ferrous and ferric iron are dissolved ions, whereas the iron compounds are considered particulate material. In order to determine the concentrations of ferrous iron, ferric iron, and general iron compounds the water samples must be collected and handled in a particular way. These samples require filtration (0.45 μm filter paper) and acidification ($\text{pH} < 2$ with Nitric Acid) in the field. Filtration separates the free ferrous and ferric ions from the particulate, solid compounds. The acidification forces the iron reactions in such a direction that preferentially the iron remains in a dissolved state – thus not accumulating on the sides of the sample bottle which would lower the iron concentration levels. Additionally, there should be no headspace left in the sample bottles as the presence of atmospheric oxygen could result in oxidation of the dissolved iron species. Once the samples are in a lab and ready for analysis, the concentrations can then be measured in a number of ways, with a: color spectrophotometer, atomic adsorption, iron chromatograph, or gas-chromatography-mass-spectrometry. Should the metal concentrations be over range for the color spectrophotometer, dilution may be required.

Objectives:

- Learn to filter and acidify samples for metals analysis.
- Learn to use a color spectrophotometer to measure total and total dissolved iron concentrations (ASTM D1068-03, APHA 3500-Fe), calculate the concentration of particulate iron, and discuss results from different water sources.

Materials:

The following materials will be used to determine the iron concentration of a water sample.

- 1 beaker
- 1 filtration setup
- Tweezers
- Filter paper
- HACH colorspectrophotometer
- FerroVer Iron power packets
- Scissors

- KimWipes
- Distilled water

Procedure:

The following procedure will be used to determine the concentrations of total, particulate, and dissolved iron for a water sample. Record the results in the data table found in **Exhibit 2**. The results will be shared with the group for discussion of variation in water quality between the different sources.

- To measure total iron:
 1. Turn on the HACH and enter program # 265 (US EPA Ferro Ver Method, Method 8008, Range = 0.02 – 3.00 mg/L).
 2. Obtain a numerically matching pair of 10 mL cuvettes. Fill both of the 10 mL cuvettes with sample water. The bottom of the meniscus should fall just about the 10 mL marker line.
 3. Place the two sample cells on the benchtop in front of the spectrophotometer. The sample cell on the left will serve for the blank and the second sample cell on the right will serve as the sample.
 4. To the second sample cell on the right, add the contents of one FerroVer powder packet to the sample cell. Hold the powder packet at the top, give it a few flicks with a finger to get the powder to the bottom of the packet, use scissors to cut off the top along the black dashed line, pinch either side of the packet and push inwards to open the packet, and invert the packet over the sample cell to dump the contents. Swirl to mix. Push the timer button to start the instrument timer for a reaction time of 3 minutes.
 5. Wipe the sides of sample cells with a KimWipe, removing any spots or smudges.
 6. Once the timer has sounded place the first, blank sample cuvette into the holding chamber of the HACH, close the lid, and hit Zero.
 7. Once a reading of 0.00 mg/L Fe is displayed, remove the blank sample cell from the HACH. Insert the second sample cuvette into the holding chamber, close the lid, and hit Read.
 8. Record your results, which are displayed in mg/L Fe, in the data table found in **Exhibit 2**.
 9. Dump the contents of the sample cell into a waste container, dump the contents of the blank into a sink, and place the cuvettes in a designated wash tray to be cleaned later by the instructor. Please be careful when handling the cuvettes as they become very slippery when wet and can be easily dropped and broken.
 10. Note: If the sample's concentration blinks at 3.0 mg/L the sample is over range and will need to be diluted. To prepare a x10 dilution, use a pipette to draw 1 mL of sample. Extrude the 1 mL of sample water into a washed and rinsed cuvette. Using the pipette with a clean tip, add 9 mL of distilled water. Both the blank and sample should be prepared in this manner. If you need a clean pair of cuvettes ask the instructor for assistance. Depending on the iron concentration of the sample – further dilution may be required. To prepare a x100 dilution, 1 mL of the x10 solution should be drawn and added to 9 mL of distilled water. To prepare a x1000 dilution, 1 mL of x100 solution should be drawn and added to 9 mL of distilled water.
- To measure total dissolved iron:
 1. To measure total dissolved iron, and then calculate the amount of particulate iron, the sample must be filtered. Typically samples are filtered ahead of time in the field, but in some cases if they are immediately taken to the laboratory after collection they may be filtered in the lab. Filtering may be done with a syringe and screw-on filter or with a larger filter assembly that is hooked up to one of a variety of vacuum systems. A filter setup has been pre-prepared for each group with a 0.45 µm filter paper

2. Turn on the vacuum pump.
 3. Pour approximately 200 mL of distilled water onto the middle of the filter. Leave the vacuum on until the entire sample has been pulled through the filter paper.
 4. Turn the vacuum off. Remove the bottom portion of the filter apparatus from the collection flask. Transfer 10 mL of the filtrate to a clean cuvette – this will serve as the “blank”.
 5. Dump the remaining filtrate and reconnect the collection flask to the filter apparatus. Turn on the vacuum pump.
 6. Pour approximately 200 mL of sample water into the middle of the filter. Leave the vacuum on until the entire sample has been pulled through the filter paper.
 7. Turn off the vacuum pump. Remove the bottom portion of the filter apparatus from the collection flask. Transfer 10 mL of the filtrate to a second clean cuvette – this will serve as the “sample”.
 8. Follow the same procedure used to measure total iron to measure the amount of dissolved iron.
 9. Record the result in data table provided in **Exhibit 2**.
- To calculate particulate iron:
 1. To calculate the amount of particulate iron, subtract the amount of dissolved iron from total iron.
 2. Record this result in the data table provided in **Exhibit 2**.

BIOCHEMICAL OXYGEN DEMAND (BOD) ANALYSIS

Introduction and Background:

The biochemical oxygen demand (BOD) test is used to determine the amount of dissolved oxygen utilized for the degradation of organic matter in waters such as wastewater effluents and polluted waters. It is one of the primary parameters used by wastewater treatment plants and various industries to determine if their treated waste stream is of acceptable quality to be pumped into a fresh surface water source like a river. This form of BOD is termed the carbonaceous demand.

BOD exertion (and utilization) is a complex process and can be affected by the following factors:

- **Microbial population:**

For degradation of the organic matter in the sample to occur, a microbial population must be present. A suitable population may already be present in the sample itself, as is the case with most wastewater samples. However, other types of water being tested may not contain a sufficient level of microorganisms on their own, requiring that the sample be “seeded” with a microbial population. In their natural environment, the seed microbial population becomes adapted to utilizing the nutrients that are available; however, when they are placed in a different environment with different biodegradable organics, it may take some time for the metabolic machinery to become adapted to the new food source. As an alternative to using a natural seed, laboratory prepared seed is available that includes a wide variety of microbial types this ensuring that biological growth of some type can occur without an acclimation period.
- **Environmental conditions:**

Factors such as pH, temperature, and availability of nutrients can affect BOD. It is important that analysis is started within 24 hours of collecting the water sample and that it is kept at 4°C until then. The pH of the sample water should lie between 6.5 and 7.5 and can be adjusted to this range with

sulfuric acid or sodium hydroxide, depending on whether the water is alkaline or acidic. Once the samples have been prepared, they should remain at approximately 20°C for the duration of the test period.

- **Type of Organic Matter:**
Organic matter such as simple sugars and amino acids can be degraded faster than polysaccharides and complex proteins, potentially influencing the rate at which oxygen is consumed.
- **Inhibiting and toxic compounds**

The organic matter that is initially utilized is classified as readily or easily biodegradable organic matter. The bacterial population is the first to utilize the organic matter, resulting in a rapid depletion of available oxygen and substrate. This results in the rapid growth of the bacterial population. As the oxygen becomes scarce, bacteria begin to die and are preyed upon by other bacteria and protozoans. The protozoans become the dominant microbe and continue until the amount of organic matter becomes negligible or the oxygen drops below a threshold level and the aerobic bacteria die. A new stage can then begin where anaerobic bacteria begin to grow (assuming that they are present at this stage in the BOD utilization cycle).

The BOD measure, in a BOD₅ determination, is a measure of the oxygen consumed in the first five (5) days of bacterial and protozoan growth. If the dissolved oxygen does not get totally consumed, then most of the growth in the experiment has been bacterial. Because the growth of biomass in the BOD experiment is highly variable, BOD determinations are often difficult to reproduce even between replicates of the same sample. BOD measures should therefore be interpreted with caution.

A different class of microbial cells called algae can also influence the results of the BOD test, especially if the sample is taken from a natural water stream such as a lake. Algae act as a source of oxygen due to their photosynthetic metabolism which produces oxygen as a by-product. To minimize the impact of algae, BOD bottles are stored in the dark during the five-day incubation period. The results of any BOD test on waters where algal growth is significant or where light is allowed to shine on the BOD bottles should be considered faulty.

The carbonaceous oxygen demand is the oxygen used by microorganisms in the breakdown of organic matter. However, oxygen may also be used to oxidize reduced forms of nitrogen. This is termed the nitrogenous demand. The oxidation of reduced forms of nitrogen requires the presence of microorganisms capable of carrying out this oxidation. In many waters, including raw sewage and primary effluent, these microorganisms are not present in sufficient enough numbers to have much influence during a 5 day BOD test. Usually, they will not exert a significant oxygen demand until day seven or eight (at 20°C). Under these circumstances, the BOD₅ measured will be the carbonaceous BOD₅ because the decrease in oxygen is attributable to bacteria using carbon compounds as a nutrient source. As the BOD test reaches the seventh or eighth day, nitrifying bacteria begin to exert a measurable amount of oxygen demand (3.76 mg O₂/mg NH₃). Measurements of BOD that include both carbonaceous and nitrogenous demand are not a true measure of the oxygen required to degrade organic matter and it is often desirable to inhibit the nitrogenous demand. Inhibition of nitrogenous demand can be achieved by using a chemical inhibitor.

BOD₅ levels in waste streams can be quite variable depending on the source of the waste and other water/wastewater quality parameters (e.g. water at lower temperatures can hold more oxygen). Animal manure may have a BOD level as high as 20,000 mg/L, sewer water is approximately 150-250 mg/L, and treated municipal sewer waste is approximately 20 mg/L.

Objectives

- To quantify the five-day carbonaceous biochemical oxygen demand (BOD₅) (ASTM D6238, APHA 5210) of a water sample and discuss results from different water sources.

Materials

The following materials will be used to determine the BOD₅ of a water sample.

- Sterile BOD bottles
- Water sample
- Sterile deionized water
- Labeling tape
- DO probe
- Squirt bottle of sterile deionized water
- Kim Wipes

Procedure

The following procedure will be used to determine the BOD₅ for a water sample. Record the results in the data table found in **Exhibit 5** and **Exhibit 2**. The results will be shared with the group for discussion of variation in water quality between the different sources.

First, each group will learn how to prepare BOD bottles for analysis. Then, the groups will analyze BOD bottles prepared 5 days ago in order to determine the sample's BOD₅. Note that we will not be diluting the samples or adding a seed or nitrogen inhibitor.

- Preparing the sample bottles:
 1. Obtain 4 sterilized BOD bottles.
 2. Prepare and adhere labels. All labels should include the Sample ID, date at $t = 0$, initials of the person(s) conducting the test, BOD day number (BOD_t, where $t = 0$ or 5), and indicate whether the bottle is the blank or sample. There should be a blank bottle and sample bottle for day 0, and a blank bottle and sample bottle for day 5.
 3. Vigorously shake the jug of sample water in order to aerate the sample, saturating the water with dissolved oxygen.
 4. Immediately upon aerating the sample water, fill the BOD bottles to the top with sample water. Place the stoppers in the bottles. When the caps are put on the bottles, they should displace water. If they do not displace water, add some more sample water. This will ensure a tight seal and prevent air from being trapped in the bottles (which could potentially alter the dissolved oxygen readings).
 5. Start a timer for 15 minutes.
 6. Vigorously shake the jug of sterile, deionized water (with the same vigor and for the same period of time as the sample water).
 7. Immediately upon aerating the deionized water, fill the blank water bottles to the top with deionized water. Place the stoppers in the bottles. When the caps are put on the bottles, they should displace water. If they do not displace water, add some more sample water. This will ensure a tight seal and prevent air from being trapped in the bottles (which could potentially alter the dissolved oxygen readings).

8. Start a second timer for 15 minutes.
9. Place all bottles for sample day 5 in the designated dark storage compartment.
10. Put the samples aside that you just prepared; you will now work with the samples prepared for you five, 5, days prior to this short course.
11. Turn on and re-calibrate the DO probe, following the instructions from the “Basic Water Quality Parameter” section above.
12. After the 15 minutes has passed for the Day 0 blank and sample bottles, measure and record the DO concentration in the data table found in **Exhibit 5**. To measure DO in the BOD bottle, Remove the stopper from the BOD bottle, carefully insert the probe into the bottle until it is at a medial depth. Holding the bottle very carefully as it is wet and very slippery, gently swirl the bottle so that water is flowing past the membrane at the end of the probe.
13. The instructor will provide each group with the DO readings for the Day 0 sample and blank of each group’s source water. Enter these values in BOD Analysis Data Table (**Exhibit 5**).
14. Measure the amount of dissolved oxygen for the prepared blank and sample bottles for the group’s sample source, for Day 5. Enter these values in BOD Analysis Data Table (**Exhibit 5**).
15. Determine the BOD₅, show your work and final result in the BOD Analysis Data Table (**Exhibit 5**):

$$\text{BOD}_t = \{(D_1 - D_2) - (B_1 - B_2)\} / P$$

Where 1= initial (t=0) and 2 = final, D = sample, B = blank, and P = dilution factor (e.g. 1mL/300mL). Note: if the sample was not diluted – 300mL sample/300mL = 1.

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Exhibit 2: Summary Data Table for Water Quality Parameter Analytical Results

Water Quality Testing Analytical Results Summary Table		
Sample ID:		
Source:		
Parameters	Results	Comments
pH		
Temperature		
Conductivity		
Turbidity		
Alkalinity		
Hardness		
Total Iron		
Particulate Iron		
Dissolved Iron		
BOD ₅		

Exhibit 4: Data Table for Hardness Analysis

Hardness	
Sample ID:	
Source:	
Initial Volume of EDTA (mL)	
Final Volume of EDTA (mL)	
Volume of EDTA used in the titration (mL) - A	
mg CaCO₃ equivalent to 1.00 mL of titrant - B	
Volume of Sample Used (mL)	
Hardness (mg/L as CaCO₃)	

Exhibit 5: Data Table for BOD Analysis

Biochemical Oxygen Demand (BOD)		
Sample ID:		
Source:		
Day 0 Results for Bottles Prepared During the Short Course		
Sample Day	Sample DO Concentration (mg/L)	Blank DO Concentration (mg/L)
0		
Pre-Prepared BOD Bottle Results		
Sample Day	Sample DO Concentration (mg/L)	Blank DO Concentration (mg/L)
0 (provided)		
5		
Calculated BOD₅ for Pre-Prepared Bottles		
BOD₅ (mg/L)		