

Qualitative Water Analysis — Student Guide

GLOSSARY

aerobic — living or occurring only in the presence of oxygen.

colorimetric — name given to a chemical test method where the result of the test is determined by the amount or the shade of the color developed.

desalination — process that removes salt from ocean waters to generate quantities of fresh water.

food web — a series of organisms through which food energy is passed.

indicator — a chemical reagent added to a test sample that shows when a chemical reaction takes place, indicated by a color change in the test sample.

polar — a bond that has an uneven distribution of charge due to the more electronegative element attracting electrons away from the less electronegative element, resulting in a molecule with a positively charged region and a negatively charged region.

potable — suitable for drinking.

precipitate — an insoluble compound formed by chemical action between two or more normally soluble compounds in solution.

qualitative — a system of analysis used to determine the presence or absence of a chemical species.

soluble — substances that will dissolve in water, such as salt and sugar.

solvent — the dissolving medium in a solution.

tailings — piles of finely ground rock discarded as a by-product of the mining process. Tailings sometimes contain heavy metals. If left exposed to forces of nature, the run-off from tailings can be toxic to terrestrial and aquatic life.

water of metabolism — water produced as a by-product of metabolic processes.

BACKGROUND

All living organisms require water to survive. In fact, water comprises 70-90% of all living organisms by mass. Within an organism, water may function as either a reactant, participating in a chemical reaction, or it may provide an important environmental component for biochemical/metabolic processes to take place. The average human adult requires 2.5 liters of water per day for proper physiologic functioning. Approximately 60% of the water that humans take in on a daily basis is through the ingestion of beverages. The balance of a human's water requirement is satisfied through the ingestion of moist food (approximately 30%), and as a by-product of oxidative metabolism (also known as the **water of metabolism**) (approximately 10%). With an ever increasing world population (1990 estimates indicate a world population size of 5 billion), there is a growing concern about the availability of fresh, quality water sources for human consumption. As one might suspect, an increase in population size results in an increase in pollution and reduced air and water quality through increased introduction of contaminants into these natural resources.

Water occupies approximately 75% of the Earth's surface. If you investigate this natural resource a little more closely, you will find that approximately 97% of the Earth's water supply is present in the form of salt water located in the planet's oceans. Due to its **saline** nature, ocean water is unfit for human consumption. The process of **desalination** has been developed to remove salts from ocean water. However, this process is currently too expensive to utilize extensively to provide large quantities of fresh water.

If 97% of Earth's water supply is present as salt water, then only approximately 3% of the Earth's water supply exists as fresh water. However, all of this fresh water is not available for human consumption and/or usage. Of the Earth's fresh water supply, approximately 2.2% is frozen in primarily continental glaciers and secondarily in valley glaciers. While scientists are currently exploring ways to exploit these fresh water sources, no efficient method has yet been identified or commercially implemented.

Most fresh water sources available to life on Earth are confined to the remaining 0.8% that exist predominantly as groundwater, secondarily as surface waters (rivers, lakes, and streams), and marginally as atmospheric moisture.

Many factors can and do affect the quality of surface and groundwater. Before a water supply may be considered **potable** — that is, safe for human consumption — this supply is evaluated for concentrations of inorganic chemicals, organic wastes, and disease-causing microorganisms.

Inorganic chemicals may be introduced into water supplies either naturally or through the actions of humans, either direct or indirectly. Water quality may be affected naturally as a function of chemical changes that result

when water moves over the surface of the land as run-off. This run-off passes over rock containing **soluble** minerals. Another source of water contamination occurs when groundwater passes through porous rock containing soluble minerals such as sodium, calcium and magnesium. Water is a **polar** molecule. The polarity of a water molecule causes this molecule to be a particularly effective **solvent**. As water passes over rock or soil containing minerals, sodium, calcium, iron, or magnesium, these substances will dissolve in the run-off and will eventually be introduced into the run-off's destined body of water. The dissolving of these soluble minerals may result in an increased concentration of sodium and chloride atoms, particularly in those areas that utilize rock salt as a mechanism for deicing roads.

Water with high concentrations of calcium and magnesium ions is known as "hard". Very hard water is undesirable for use in industry, as it results in the production and build-up of scale, rock-like mineral deposits which line pipes and boilers, eventually leading to reduced operating efficiency and costly replacement of equipment. You may have seen evidence of scale on faucets or shower heads. If you have stood in a room with a hot water tank as the tank was heating, you may also have unknowingly heard evidence of scale build-up. As the tank heats, a noise like rocks or pebbles being boiled is most likely attributable to the presence of scale on the inside of the boiler.

On the other hand, water that is low in dissolved minerals is known as soft water. One characteristic of soft water is that less soap is required to produce a rich lather. In fact, generally, the softer the water, the richer the lather. You can see this for yourself if you wash your hands using equal quantities of soap, first in tap water and then in deionized water. Soft water usually has a lower pH than hard water, and is mildly acidic to very acidic. Very soft waters are usually treated with lime, a base, resulting in a neutralization reaction, which increases the pH of the water and makes it less corrosive. This same process of dissolving and subsequently carrying off of soluble minerals occurs as run-off from precipitation passes through mine **tailings** containing potentially toxic elements such as mercury and lead. Inorganic chemicals may also be introduced into surface and groundwater sources, either directly through dumping by industry, or indirectly, as in the case of agricultural run-off containing pesticides.

After toxic substances have been introduced into an aquatic system, they make their way into the **food web** as aquatic plants and algae (producers) absorb the heavy metal toxins. The concentration of toxins becomes magnified in the tissues of organisms throughout the food web as primary consumers eat large amounts of plants and algae, and accumulate toxins in their tissues. The consumption of many primary consumers by larger secondary consumers further magnifies the concentration of toxins at higher energy levels within a food web. The process by which the concentration of toxic substances increases from one energy level to the next through the energy pyramid is known as **bioaccumulation** or **biologic magnification**. This process has been attributed to mercury poisoning and related health effects observed in human populations that consume large amounts of fish from polluted waters. One example of this can be found in fish from the Great Lakes, located in the northeastern region of the U.S. Although the Great Lakes are monitored for levels of pollution on a regular basis and are, in fact, cleaner than they were 10-20 years ago, the amount of bioaccumulation of some toxins in some game species of fish in Lakes Erie and Ontario have warranted a special advisory by the New York State Department of Health (DOH) to limit consumption of specific fish species known to have elevated contaminant levels as determined by federal food standards. The DOH recommends that "women of childbearing age, infants, and children under the age of 15 should not eat fish with elevated contaminant levels".

Organic materials or organic wastes are substances produced by plants and animals, canning, brewing, meat packing industries, and paper mills. The introduction of these wastes into waterways and bodies of water adversely affect aquatic life, as **aerobic** bacteria naturally present in these waters use oxygen normally utilized by fish and other aquatic organisms to decompose these organic wastes. In environments with heavy organic waste contamination, the oxygen content of the water may be greatly reduced resulting in the death of fish and other aquatic organisms.

The presence of large amounts of organic wastes, particularly nitrates and phosphates from fertilizers, pesticides, or detergents, may also cause increased plant and algae population growth. This increased growth results in dense layers of algae which form at the surface of lakes and ponds. Algal organisms and plants found below the uppermost layer soon become light and/or oxygen deprived and subsequently die, adding material to the bottom of the lake. Again, as bacteria decompose these organisms, the oxygen content of the water that is available for aquatic life becomes greatly reduced.

A third class of pollutants examined when evaluating the quality of a water source is the presence of disease-causing microorganisms. These microorganisms may be present if the water has been contaminated by untreated sewage and wastes. Disease causing microorganisms such as *Escherichia coli*, infectious bacteria, and viruses are found in the intestines of warm blooded animals and will be introduced into aquatic systems if the metabolic wastes from these organisms are left untreated prior to entering the water supply. Chlorine is routinely used as a disinfectant in the treatment of public water supplies, eliminating or significantly reducing the potential biologic hazard that an untreated water supply poses.

In the following laboratory investigation you will collect water samples and conduct a **qualitative** analysis of these samples, to determine the presence of inorganic substances (hard water, pH estimate, elemental chlorine and iron) and organic substances (nitrates, phosphates, ammonia, and sulfate).

LAB OBJECTIVE

- To test collected water samples for the presence of common industrial/biologic contaminants and mineral concentration as indicated by hard water and pH tests.

LAB PROCEDURE

This lab consists of several tests that you will perform using a portion of the water sample you collected and the chemicals provided. Each test is set up at a separate lab station in the classroom. Place your microfuge tubes in your tray, and carry the tray from station to station. Your group should start analyzing your water sample at any lab station (your teacher may designate one for you). Be sure to record all results for each test in the appropriate space in Data Table I in the Results section of the worksheet (page S5). Following completion of all tests, answer the Worksheet questions, as instructed by your teacher.

NOTE: Before removing water from your testing sample, shake the vial, to create a homogeneous solution. This should be done before performing each of the following tests.

Nitrate Station

- Label a clean, dry microfuge tube "nitrate" or " NO_3^- ".
- Pipet 0.5 mL of water from your sample vial into the clearly labeled microfuge tube.
- Using the 0.1 g scoop provided at the lab station, place a half filled scoop of cadmium powder in the microfuge tube containing 0.5 mL of your water sample. Close the lid of the tube and shake well.
- Add 0.5 mL sulfanilic acid (fill the microfuge tube to 1.0 mL line) to your water sample in the microfuge tube containing cadmium powder. Close lid and shake well.
- Using a clean pipet, add 5 drops of chromotropic acid to the water sample containing the other two reagents. Close the lid of the vial and shake well. Set this sample aside and record your results at end of class period. The formation of a dark red complex is a positive test result for the presence of nitrate. At the end of class, record your results for this test in the appropriate space in Data Table I, writing either (+) to indicate the presence of nitrate or (-) to indicate the absence of nitrate.

Iron Station

- Label a clean, dry microfuge tube "iron" or "Fe".
- Pipet 0.5 mL of water from your sample vial into the clearly labeled microfuge tube.
- Using a clean pipet, add 5 drops sulfuric acid to the water in the microfuge tube. Close the lid on the tube, and shake the tube well.
- Using the 0.1 g scoop provided at the lab station, place a scoop of ammonium thiocyanate crystals into the microfuge tube containing your water sample and sulfuric acid. Close the lid on the tube, and shake the tube well. The formation of an orange or dark purple solution is a positive indicator for the presence of iron. Record your results for this test in the appropriate space in Data Table I, writing either (+) to indicate the presence of iron or (-) to indicate the absence of iron.

Phosphate Station

- Label a clean, dry microfuge tube "phosphate" or " PO_4^- ".
- Pipet 0.5 mL of water from your sample vial into the clearly labeled microfuge tube.
- Using a clean pipet, add 5 drops **sulfuric acid** to the water sample in the microfuge tube. Close the lid on the tube, and shake the tube well.
- Using a clean pipet, add 5 drops of ammonium molybdate to the water sample in the microfuge tube. Close the lid on the tube, and shake the tube well.
- Using a clean pipet, add 5 drops ascorbic acid to the water sample in the microfuge tube. Close the lid on the tube, and shake the tube well. Wait several minutes for the reaction to take place. The formation of a blue solution is a positive indicator for the presence of phosphate. Record your results for this test in the appropriate space in Data Table I, writing either (+) to indicate the presence of phosphate or (-) to indicate the absence of phosphate.

Chloride Station

- Label a clean, dry microfuge tube "chloride" or "Cl".
- Pipet 0.5 mL of water from your sample vial into the clearly labeled microfuge tube.
- Using a clean pipet, add 0.5 mL silver nitrate to your water sample in the microfuge tube. Close the lid on the tube but **DO NOT SHAKE THE TUBE**. The formation of a white precipitate is a positive indicator for the presence of chloride ions. Record your results for this test in the appropriate space in Data Table I, writing either (+) to indicate the presence of chloride ions or (-) to indicate the absence of chloride ions.

Ammonia Station

1. Label a clean, dry microfuge tube "ammonia" or " NH_4 ".
2. Pipet 0.5 mL of water from your sample vial into the clearly labeled microfuge tube.
3. Using a clean pipet, add 5 drops potassium hydroxide (KOH) to your water sample in the microfuge tube. Close the lid on the tube and shake the tube well.
4. Using the pipet provided, add 3 drops Nessler reagent to the water sample in the microfuge tube. Close the lid on the tube but **DO NOT SHAKE THE TUBE**. The formation of a dark orange precipitate is a positive indicator for the presence of ammonia. Record your results for this test in the appropriate space in Data Table I, writing either (+) to indicate the presence of ammonia or (-) to indicate the absence of ammonia.

Hard Water Station

1. Label a clean, dry microfuge tube "hard water" or "HW".
2. Pipet 0.5 mL of water from your sample vial into the clearly labeled microfuge tube.
3. Using the pipet provided, add 5 drops ammonium hydroxide (NH_4) to your water sample in the microfuge tube. Close the lid on the tube and shake the tube well.
4. Using the pipet provided, add 1 drop of the indicator solution, Eriochrome Black T, to your water sample containing NH_4 . Close the lid on the tube and shake the tube well. The solution should be purple in color at this point.
5. Using the pipet provided, add EDTA to the tube, until the total volume of the solution in the tube is 1.0 mL, as indicated by the markings on the side of the microfuge tube. The formation of a blue solution is a positive indicator for the presence of hard water. Record your results for this test in the appropriate space in Data Table I, writing either (+) to indicate the presence of hard water or (-) to indicate the absence of hard water.

Sulfate Station

1. Label a clean, dry microfuge tube "sulfate" or " SO_4 ".
2. Pipet 0.5 mL of water from your sample vial into the clearly labeled microfuge tube.
3. Using the pipet provided, add 3 drops of barium chloride to your water sample in the microfuge tube. Close the lid on the tube but **DO NOT SHAKE THE TUBE**. The formation of a white precipitate is a positive indicator for the presence of sulfate. Record your results for this test in the appropriate space in Data Table I, writing either (+) to indicate the presence of sulfate or (-) to indicate the absence of sulfate.

Chlorine Station

1. Label a clean, dry microfuge tube "chlorine" or "Cl".
2. Pipet 0.5 mL of water from your sample vial into the clearly labeled microfuge tube.
3. Using the pipet provided, add 0.5 mL o-Tolidine to your water sample in the microfuge tube. Close the lid on the tube and shake the tube well. The formation of a yellow colored solution is a positive indicator for the presence of chlorine. Record your results for this test in the appropriate space in Data Table I, writing either (+) to indicate the presence of chlorine or (-) to indicate the absence of chlorine.

pH Station

1. Label a clean, dry microfuge tube "pH".
2. Pipet 0.5 mL of water from your sample vial into the clearly labeled microfuge tube.
3. Using the pipet provided, add 2 drops universal indicator solution to your water sample in the microfuge tube. Close the lid on the tube and shake the tube well. Using the information provided below record approximate pH of your water sample in appropriate space in Data Table I.

pH color scale for Universal Indicator

pH	Color transitions
2	red
3	red/orange
4	orange/red
5	orange
6	yellow
7	yellow/green
8	green
9	green/blue
10	blue