

Introduction to the Chemistry Laboratory
pH, Chlorine Content and Alkalinity of Water

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pH, Chlorine Content, Total Alkalinity, Acid Demand

Purpose: In this experiment, you will become familiar with the measurement of pH, Chlorine and Alkalinity/Hardness of water as used in drinking water, swimming pools and hot tubs.

- **Run all 4 tests on ONE sample prior to going to the next sample.**
- **Test 2 uses the solutions from Test 1, so don't throw them out!**

Swimming Pool and Drinking Water is treated chemically in order to remove and keep harmful bacteria out. An oxidizing agent such as Chlorine, Bromine or Ozone is added in a very small amount to prevent the growth of bacteria and algae. In a home swimming pool or hot tub, those in charge of maintenance must measure the concentration of the oxidizing agent and add the appropriate chemicals to maintain the proper balance to prevent the growth of bacteria and algae and prevent corrosion. Chemicals also may be added to preserve the neutral pH [how acid or basic it is]. Proper chemical balance prevents corrosion of metal tubes, pumps and fittings.

All of the parameters discussed below can and will be measured using a readily available Swimming Pool Test Kit. The chemicals in the solutions in the Basic 4 Test Kit that is used in this lab are:

Solution #1: "OTO reagent" consists of Otolidine Dyhydrochloride and muriatic acid.

Solution #2: "pH liquid reagent" consists of phenol red and baking soda [Sodium Bicarbonate buffer].

Solution #3: Consist of hydrochloric acid at a known concentration.

Solution #4: Consist of sodium thiosulfate.

Solution #5: Consist of bromphenol blue in a alcohol solution.

1. pH is a measure of how Acidic or Basic the water is.

Acidity is a measure of the amount of Hydrogen Ion [H^+] concentration and is measured in units of pH. pH is measured as minus the Log of the Hydrogen Ion concentration " $-\text{Log} [H^+]$ " and varies from 1 to 14. pH of 1 is very acidic and pH of 7 is neutral, pH of 14 is very basic

Basicity is a measure of the amount of Hydroxyl [OH^-] present. We usually do not measure this value.

An acidic pH of 1 - 2 or a basic pH of 12 - 14 can be harmful to living tissue and will react with most metallic parts such as pipes, pumps and containers. Concentrated Hydrochloric Acid [HCl] with a pH of 1-2 will react with Iron Metal to form Iron Chloride and Hydrogen Gas - it will dissolve the Iron. HCl will also react with and dissolve concrete, so don't spill it on your pool deck. Concentrated Sodium Hydroxide [$NaOH$] with a pH of 12 - 14 will react with Human Skin turning it into a soap like material. Drano, the common plumbing cleaner, consists of $NaOH$. Muriatic Acid is used to control the pH of pools.

For most household uses, we like to keep water in a pH range of 6 - 8. Again, a pH of 7 is neutral.

Due to various equilibrium reactions, it can be difficult to keep a sample of water [e.g. a 10,000 gallon swimming pool] at a pH of 7. Under normal circumstances it would take very little acid or base to change the value from a neutral pH of 7. A slight amount of "Acid Rain" (Carbon Dioxide dissolves in water to produce Carbonic Acid H_2CO_3 , a weak acid) would lower the pH out of the desired 7.2 -> 7.8 range. We control this by adding chemicals such as Sodium Bicarbonate [$NaHCO_3$] that will act as a buffer. This aids in absorbing or reacting with excess acid or base to keep the pH in the desired range.

2. Acid Demand is a measure of how much acid, Muriatic Acid which is a 31% solution of HCl and is commonly used for swimming pools, needs to be added to your water [swimming pool] in order to return the pH to a near neutral measurement. Muriatic Acid will dissolve concrete, so don't spill it on your deck.

3. Chlorine Content is a measure of the amount of free chlorine in the pool. Chlorine kills bacteria and algae. You don't really need to measure the Bromine Content of your sample.

4. Total alkalinity is a measure of the amount of alkaline salt buffering agents is present in the sample. Total Alkalinity is the capacity of a water sample to neutralize any acid or base that was added. The pH of the sample will remain approximately in the 7.0 -> 7.9 range even though acid or base is added.

Test #1. pH [how acidic or basic is the sample]

- 1. Rinse out at least once and refill** the Large CLEAR side of the measuring tube with a water sample to the proper mark. This is the side next to the Red / Orange color marks.
- 2. Add 1 drop of Solution #4 (Green Top).** Place the cap on the sample vial top and **mix by inverting the tube slowly once, do not shake it.** This solution removes the Chlorine from the sample. The presence of Chlorine will affect the color used to measure the pH and so it is removed from the sample. The chemicals need to be mixed prior to adding the next reagent so the Chlorine is removed from the sample.
- 3. Add 5 drops of Solution #2 (Red Top),** the Red Indicator. This is the pH indicator dye.
4. Place the cap on the vial top and **mix by inverting the tube slowly once, do not shake it.**
- 5. Read the pH** of the water from the color comparison on the side of the vial.

Note:

pH 3 and below is VERY Acidic

pH 7 is Neutral

A pH of 7.2 -> 7.8 is the desired range for swimming pools

pH above 10 is Very Basic.

- 6. Go To Test 2. Save and use this sample for the "Test 2 - Acid Demand" test in the next section.** Run the pH and then the Acid Demand on the same sample while it's still in the plastic measuring tube.

Test #2. Acid Demand

If the **pH of the water sample is higher than 8.0**, then acid must be added to the swimming pool to lower the sample's pH. We will use this test to determine how much acid must be added to the swimming pool.

If the pH is 7.5 or less, DO NOT run this test, it will not work.

Remember, pH of 1 is very acidic, pH of 7 is desired and pH of 14 is very basic. We can measure how much acid is required to be added to the swimming pool using the "Acid Demand" test. Using the same sample from the pH test above, we will add drop wise amounts of a calibrated acid solution until the pH of the solution is in the desired range of 7.4

- To the solution from #1 above, add one drop of Solution #3 and gently **mix by inverting the tube slowly once, do not shake it**.
- Read the pH of from the Red / Orange color chart. If the pH is still ABOVE 7.4, go back to step 1. Do Not count "The Drop" that lowers the pH to 7.2 or below.
- Refer to the Acid Demand Table below looking up the number of drops of Solution #3 added to bring the pH to 7.4. **Assume the pool size is 15,000 gallons.** Determine how much Muriatic Acid [31% Hydrochloric Acid - HCl] is required to be added to bring the pH into the appropriate value.

If you were really adding the acid to a pool, you should wait a time period of several hours with the pool pumps running for all of the acid to mix properly with the pool water to stabilize the pH. The stabilization reaction is very fast, but the acid must be mixed with all of the pool water. You would then rerun the tests.

The Muriatic Acid used for swimming pools, in general is harmless to most people's skin. It can hurt an open sore, it can blind you if it gets in your eyes, and it can kill you if ingested. But, unlike other acids, Muriatic Acid will not usually harm skin or clothes. It will react with most metals. It will dissolve [react with] concrete such as pool decks and should not be spilled on a cement deck. Stomach acid consists of dilute Hydrochloric Acid and is at a pH of 1 – 2.

Amount of Muriatic Acid to be added to the pool.

| Pool Capacity in Gal | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 |
|--|----------|----------|-----------|----------|----------|-----------|-----------|----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|
| 1,000 | 4/5 | 1 3/5 | 2 2/5 | 3 1/5 | 4 | 4 4/5 | 5 3/5 | 6 2/5 | 7 1/5 | 8 | OZ | | | | |
| 10,000 | 1 C | 1 P | 1.5 P | 1 Q | 2.4 P | 3 P | 3.5 P | 2 Q | 4.5 P | 2.5 Q | 5.5 P | 3 Q | 3.25 Q | 3.5 Q | 1 G |
| 15,000 | 1.4 C | 1.5 P | 2.15 P | 3 P | 2 Q | 2.25 Q | 2.5 Q | 3 Q | 3.5 Q | 1 G | 1 G | 4.5 Q | 1.25 G | 1.25 G | 1.5 G |
| 30,000 | 1.5 P | 3 P | 2.25 Q | 3 Q | 1 G | 4.5 Q | 1.25 G | 1.5 G | 1.75 G | 2 G | 2 G | 2.25 G | 2.5 G | 2.75 G | 2.75 G |
| Oz = oz of Muriatic Acid, C = Cup, P = Pint, Q = Quart, G = Gallon | | | | | | | | | | | | | | | |

Test #3. Free and Combined Chlorine Content

1. **Rinse and Fill the Small CLEAR** side of the measuring tube with a water sample to the proper mark. This is the side next to the Yellow color marks
2. **Add 3 drops of the Solution #1**, the Yellow Chlorine indicator.
3. Place the cap on the vial top and **mix by inverting the tube slowly once, do not shake it**.
4. **Read the Chlorine content** from the color comparison within 2-3 seconds and compare it to the colors on the tube. This is the amount of "Free Chlorine" in the sample in Parts Per Million (PPM).
5. **Wait 2 minutes** and **re-read the Chlorine content** from the color comparison. This is a measure of the amount of "Free Chlorine" and "Combined Chlorine". "Free Chlorine" is readily available to react or oxidize to kill bacteria and Algae. "Combined Chlorine" is chemically bonded to other atoms in the form of a compound and must be broken down to "Free Chlorine" to be useful.

The desired Chlorine level is between 1.0 - 2.0 ppm. Chlorine levels below 0.5 require the addition of addition of more chlorine to the water. Levels above 3.0 are higher than required to preserve the water and may actually approach the level where the chlorine can attack [Bleach out] cloth materials.

Test #4. Total Alkalinity

1. **Rinse and Fill** the Large CLEAR side of the measuring tube to the proper mark with a water sample. This is the side next to the Red / Orange color marks.
2. Add 1 drop of Solution #4 [Green Top], and **mix by inverting the tube slowly once, do not shake it**. Solution #4 contains Sodium Thiosulfate which is used to get rid of the free Chlorine. Free Chlorine can interact with the other dye use in this test.
3. Add 1 drop of Solution #5 [Purple Top], and **mix by inverting the tube slowly once, do not shake it**. Solution #5 contains the pH sensitive dye Bromphenol Blue. This will turn yellow at and below a pH of 3.0 and purple / blue above pH 4.6. The test solution should be a Violet / Purple / Blue color.
4. Add Solution #3 [Calibrated Hydrochloric Acid], one drop at a time. **Mix by inverting the tube slowly once** after each drop is added. Continue adding until the color makes a permanent change to yellow or clear.
5. Multiply the number of drops of the solution added by 10 to determine the total alkalinity. E.g.: 5 drops = 50 ppm.
6. If the Total Alkalinity is less than 80, then additional Sodium Bicarbonate will be needed to be added to the source of the sample [e.g. 20,000 gallon swimming pool].
7. If the Total Alkalinity is more than 100, then additional acid needs to be added to the pool to react with and remove the bicarbonate buffer and the above parameters closely monitored.