Experiment 8.
Alkalinity of a Water Resource

Experimental Procedure

Objectives

- To Properly obtain a water sample
- To determine two levels of alkalinity in a water sample and to explain their differences

Introduction

Alkalinity

Alkalinity is a measure of the buffering capacity of the water.

Most of the buffering action in natural waters due to *primarily* to the presence of **carbonates and bicarbonates**.

$$H_2CO_3(aq) + H_2O(l) \rightleftharpoons H_3O^+(aq) + HCO_3^-(aq) \quad K_{a1} = 4.3 \times 10^{-7}$$

$$HCO_3^-(aq) + H_2O(l) \rightleftharpoons H_3O^+(aq) + CO_3^{2-}(aq) \quad K_{a2} = 4.8 \times 10^{-11}$$

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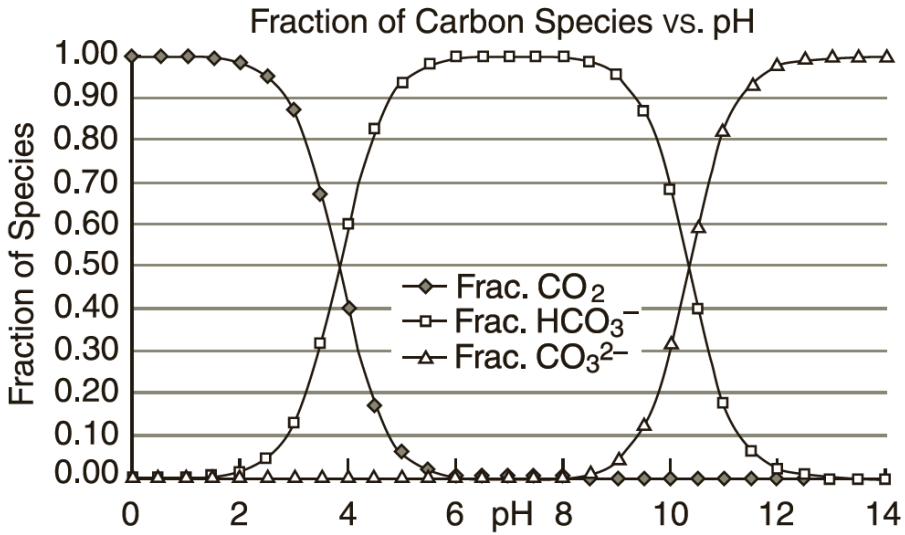


Experimental procedure

Overview

A water sample is obtained from either a natural or potable source of water. At least three samples (Filtered if necessary) of the water are titrated to the phenolphthalein endpoint and, again, to the methyl orange endpoint to determine the "P" and "T" alkalinities, respectively, for a total of six (minimum) titrations. Calculations with the data are to present the results in units of ppm $CaCO_3$. Be aware of the number of significant figures when recording data.

Part A. A Standard 0.015 M HCl Solution

1. Preparation of an HCl solution

Prepare 500 mL of a ~0.015 M HCl solution, starting with 1 M HCl. Show the calculations on your *Report Sheet*.

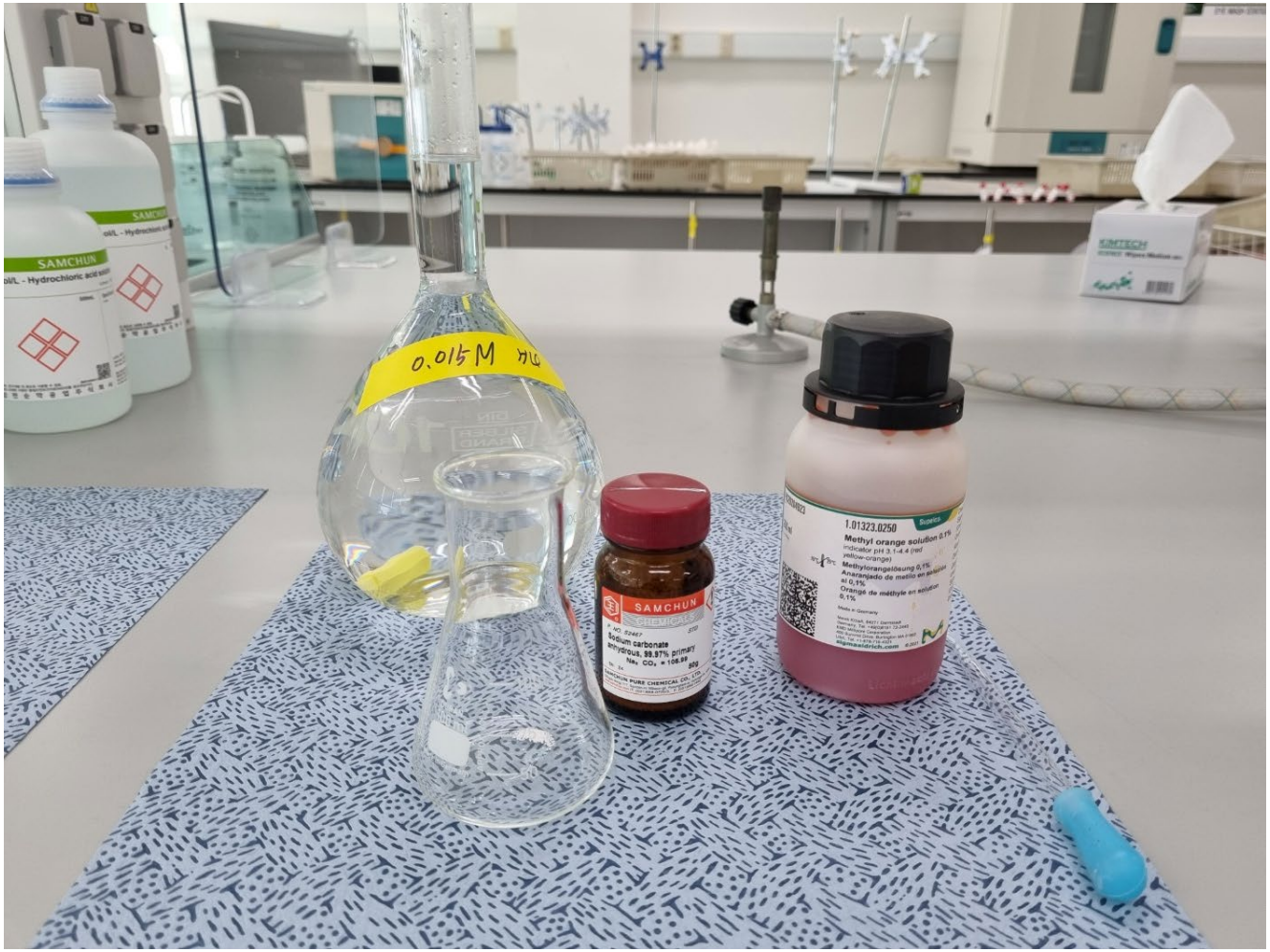
2. Samples of Na_2CO_3(s), a primary standard

a. Calculate the mass of Na_2CO_3 (molar mass = 105.99 g/mol) that is neutralized at the methyl orange endpoint with about 25 mL of 0.015 M HCl (show the calculation on your Report Sheet- this is a small mass!). Use weighing paper to measure the tared mass (±0.001 g) of at least three samples of anhydrous Na_2CO_3 (previously dried at 100°C) based on your calculations.

b. Transfer each sample to a clean, dry 125- or 250-mL Erlenmeyer flask, dissolve with ~25 mL of deionized water, and add several drops of methyl orange indicator.

c. Place a white sheet of paper beneath the Erlenmeyer flask.

3. Prepare the HCl titrant. Prepare a clean 50-mL buret for titration. Rinse the buret with 3-5 mL of the HCl solution, roll the buret to rinse the wall, and drain through the buret tip. Fill the buret with the HCl solution, and drain the tip of air bubbles; after 10-15 seconds, read and record the volume using all certain digits plus one uncertain digit.


SAMCHUN
0.015M
SAMCHUN
Sodium carbonate
anhydrous, 99.97% primary
1.01323.0250
Methyl orange solution 0.1%
KIMTECH

4. Titrate to standardize the HCl solution.

Slowly add the HCl titrant to the Na_2CO_3 solution. As the color change slows with the addition of titrant, gently warm the Erlenmeyer flask on a hot plate for 1-2 minutes to evolve any of the CO_2(aq) that has formed during the titration.

Continue to titrate the solution to the methyl orange endpoint. Read and record the volume of the HCl solution in the buret.

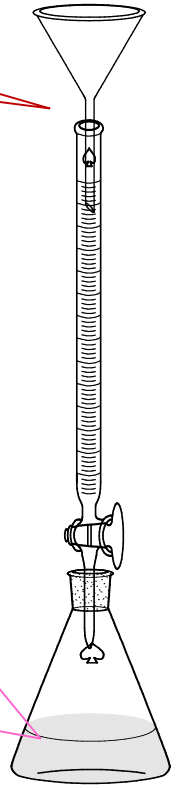
5. Additional Trials

Repeat the analysis for the two remaining samples.

6. Calculations

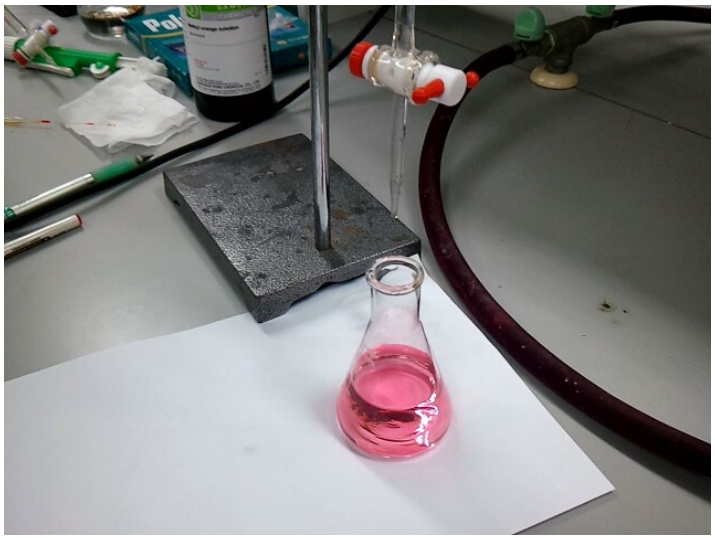
Calculate the molar concentration of HCl for each sample and the average molar concentration. Show a sample calculation on your *Report Sheet.*

~0.015M HCl solution



Standard Sodium Carbonate Solution + 25 mL of distilled water+ Methyl orange indicator (several drops)


0.01N



PART B. Preparation of Water Sample

1. Select and prepare the water sample. Select a site for securing a water sample-one recommended by instructor or by the student water chemist. Use a clean 500-mL Erlenmeyer flask to obtain a water sample. If the sample is from a natural source (i.e., stream, lake, river, *etc*), gravity filter the sample before proceeding to Parts C and D.

PART C. Determination of “P” Alkalinity

1. **Prepare the sample for titration.** Label three, clean 125-mL Erlenmeyer flasks for the "P" alkalinity analysis. Pipet 25.0 mL of the water sample (filtered if necessary) into each of the flasks and add 2-3 drops of phenolphthalein. Place a white sheet of paper beneath the receiving flask.

2. **Prepare the HCl titrant.** Prepare a clean buret. Add 3-5 mL of the standard HCl solution to the buret, roll the solution to wet the wall of the buret, dispense through the buret tip, and discard. Use a clean funnel to fill the buret-dispense a small portion through the buret tip to remove air bubbles. Read and record the volume of HCl solution in the buret according to Technique 16A.2, using all certain digits plus one uncertain digit.

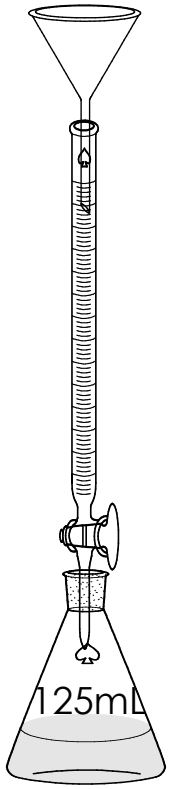
3. **Titrate for "P" alkalinity.** Slowly dispense the HCl titrant into the water sample. Swirl the flask as titrant is added(Technique 16C.4). As the color fade of the phenolphthalein slowly decreases and the return to pink slows, add the HCl titrant dropwise. When on drop (ideally, half-drop) results in the disappearance of the slightly pink color, stop the titration, and again (after ~ 15 seconds) read and record the volume of titrant in the buret.

4. **Additional trials.** *Repeat the "P" analysis on at least two more samples.*

5. **Calculations.** *Calculate the "P" alkalinity for each sample and the average "P" alkalinity, expressed in ppm $CaCO_3$.*

Water Sample 1: Evian or ..

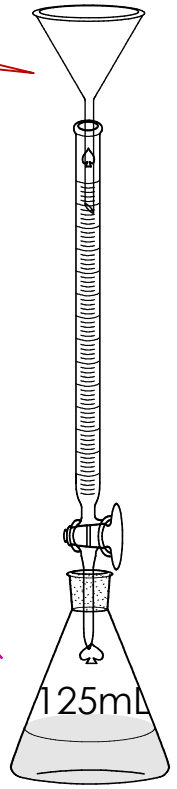
Standardized HCl solution



Water sample 25.0 mL
+ phenolphthalein indicator (2 ~ 3 drops)

Water Sample 2: Unknown sample

Standardized HCl solution



1 mL of Water sample 2 + Distilled water 25.0 mL
+ phenolphthalein indicator (2 ~ 3 drops)

PART D. Determination of “T” Alkalinity

1. **Prepare the sample for titration.** *Repeat PART C.1, substituting methyl orange indicator for phenolphthalein.*

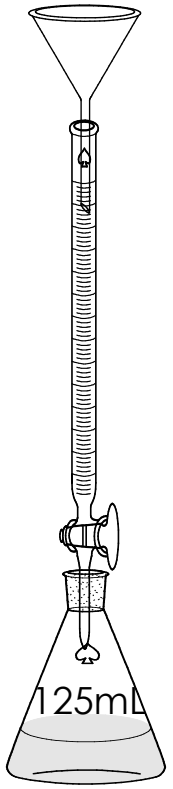
2. **Analysis for "T" alkalinity.** *Repeat PART C.2 if necessary. Slowly add the HCl titrant to the water sample. As the rate of the color change decreases with the addition of titrant, gently warm the Erlenmeyer flask on a hot plate for 1-2 minutes to evolve any of the $CO_2(aq)$ that has formed during the titration. Continue to titrate the solution to the methyl orange endpoint (see footnote 2). Read and record the volume of the HCl solution in the buret.*

3. **Additional trials.** *Repeat the "T" analysis on at least two more samples.*

4. **Calculations.** *Calculate the "T" alkalinity for each sample and the average "T" alkalinity, expressed in ppm $CaCO_3$. Additionally, calculate the standard deviation and the relative standard deviation for the "T" alkalinity.*

Water Sample 1: Evian or ..

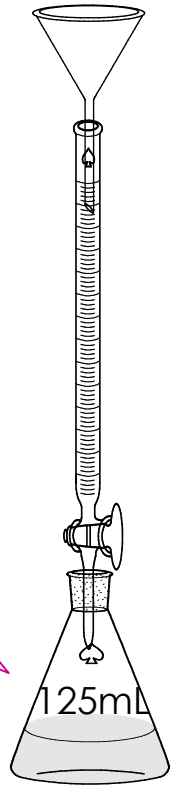
Standardized HCl solution



Water sample 25.0 mL
+ methyl orange indicator (2 ~ 3 drops)

Water Sample 2: Unknown sample

Standardized HCl solution



1 mL of Water sample 2+Distilled water 25.0 mL
+ methyl orange indicator (2 ~ 3 drops)

Using of Buret

Cleaning the Buret

In order for your buret to perform optimally, it must be properly cleaned. To clean the buret, use the following procedure:

1. Rinse with distilled water:
With the stopcock closed, add some distilled water to the buret. Tip and roll the buret, allowing the water to have contact with all of the inside surfaces. Open the stopcock and allow the water to drain. If the water drains without leaving any droplets on the side, repeat the rinse twice more then move to step two. If droplets remain on the inside surface, wash the buret with detergent solution, rinse several times with tap water, then rinse three times with distilled water.

2. Rinse with solution:
After draining the final distilled water rinse, close the stopcock and add about 5 mL of the solution to be dispensed from the buret. Again, roll and tip the buret so the solution has contact with all the inside surfaces. Open the stopcock and allow the solution to drain. Repeat this twice more. Discard the solution used in the rinses.

After you are finished with the buret in your experiment, rinse it by filling it with distilled water and allowing it to drain.

Loading the Buret

Once the buret is clean, clamp it to a stand using a buret clamp. Always make sure the burette is clamped in a perfectly vertical position before taking any readings.

When adding solutions to the buret, make sure the stopcock is closed (horizontal position). Unclamp the buret and tilt it slightly while pouring the solution slowly down the inside surface. This will prevent the formation of air bubbles.

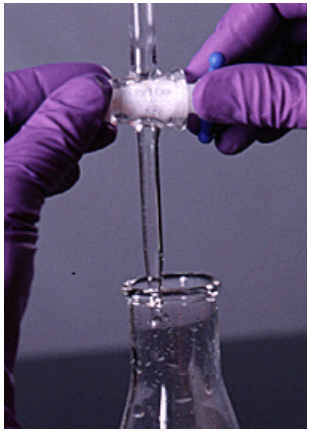
1. Get the appropriate amount of solution in a clean, dry beaker.

2. Pour a few milliliters of solution into the buret. Open the stopcock all the way in order to force all the air out of the stopcock and tip. Close the stopcock before the solution drains below the stopcock. If the tip still contains air, add a few more milliliters of solution and repeat the process. Repeat this until you are convinced no more air is left in the stopcock or tip. Discard the solution that you have run through the buret.

3. Using the procedure described above for adding solutions to the buret, fill it to a level just above 0.00 mL. Drain the buret to just under 0.00 mL. This will properly form the meniscus. DO NOT ATTEMPT TO ADJUST THE MENISCUS TO EXACTLY 0.00 mL. THIS IS AN INCREDIBLE WASTE OF TIME.

4. Touch the tip of the buret to the inside wall of a beaker in order to remove any drops on the tip. Do not wipe the tip. Wait a few seconds for the solution to drain to the top of the fluid level, then record the initial buret reading in your notebook.

5. Loosely cover the top of the buret with a cocked, small beaker or a loosely fitting piece of aluminum foil. This will keep dust out of the buret.



Reading the Buret

In order to make the meniscus easier to see, place a white card with a black mark on it behind the buret. Align the black mark so that it is just under the meniscus.

1. Get your eye level with the bottom of the meniscus. Looking up or down on the meniscus will cause a parallax error.

2. Read the buret to the nearest 0.01 mL. The marks occur every 0.1 mL, so the last number will have to be an estimate. With practice, you should be able to do this quite accurately.

Operating the Buret

Proper buret technique is an important laboratory skill that may take some practice to develop. Although is may seem initially awkward, a right handed person should operate the buret with the left hand, and a left handed person should operate the buret with the right. This leaves your more coordinated hand to swirl the reaction flask if needed.

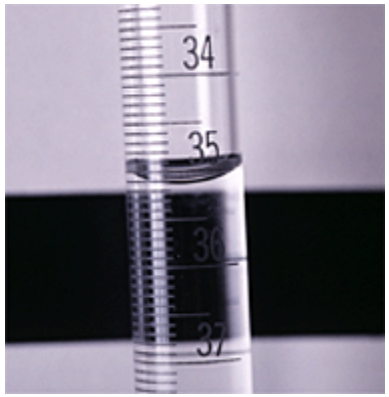
1. Before delivering any solution, record the initial buret reading in your notebook.

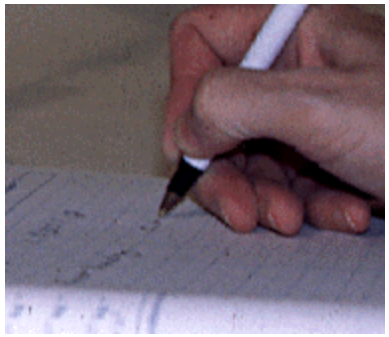
2. Open the stopcock by twisting it 90 degrees into the vertical position and allow the solution to drain. As you near the desired volume, slow the flow by turning the stopcock back toward the closed position. You should be able to control the buret to deliver one drop at a time. When the desired volume has been delivered, close the stopcock.

3. Wait a couple of seconds, then record the final buret reading.

4. Calculate the volume delivered by subtracting the initial reading from the final reading: initial reading - final reading

When delivering solutions, you must not allow the solution to drain below the bottom of the calibration range. If this is about to occur, close the stopcock and take a final reading. Refill the buret, get an initial reading, and continue delivering solution. The total volume delivered is the sum of the volume delivered the first time and the volume delivered the second time.





Cleanup

Discard the HCl solution remaining in the buret as directed by TA. Rinse the buret twice with tap water and twice with deionized water, discard each rinse through the buret tip into the sink.