Lab 7. Analysis of Hard Water

Prelab Assignment

Before coming to lab:

- Use the handout "Lab Notebook Policy" as a guide to complete the following sections of your report for this lab exercise *before* attending lab: Title and Date of Lab, Introduction, Materials/Methods and Data Tables. An outline or flow chart of the procedure is appropriate for the Materials/Methods section. Ensure that the table of contents of your lab notebook is current.
- Calculate how many grams of Na_2EDTA dihydrate will be needed to prepare 500.0 mL of an EDTA solution that is approximately 0.010 M. Record your calculation in your lab notebook.
- Complete the Prelab questions on the last page and hand in at the start of lab or when instructed by your instructor.
- Read page 4, "How to use Volumetric Pipettes and Burets"

Purpose

In this laboratory you will first learn to prepare a solution of EDTA and standardize it by titration. You will then use the same titration techniques to measure the hardness of two water samples, one an unknown solution prepared by the lab personnel, and the other will be one you bring from home. You will receive an additional grade dependent on your accuracy in determining the hardness of the unknown sample, as explained in the course syllabus.

Introduction

People who live in other regions of the country, such as the Midwest, are very familiar with "hard water." Soap doesn't lather well in hard water, and there is a constant build up of a ring of soap scum in the bathtub that has to be scrubbed to remove. What is hard water, and why does it have this effect? ***Hard water*** contains a higher than normal concentration of calcium and magnesium ions. These ions form precipitates with soap, causing the build up of soap scum. Additionally, since the soap molecules are being precipitated by the Ca^{2+} and Mg^{2+} ions, there is less soap available to form lather. Taking a shower in hard water can be very frustrating!

Another effect of hard water is "boiler scale." When hard water comes into contact with dissolved carbonates, a precipitate of insoluble calcium carbonate can form. This "scale" can build up on the inside of water pipes to such a degree that the pipes become almost completely blocked.

The following chart shows how hard water is classified. For reporting purposes, hardness is reported as parts per million (ppm) $CaCO_3$. In other words, even though both Ca^{2+} and Mg^{2+} contribute to water hardness, it is reported as though all hardness ions are Ca^{2+} from $CaCO_3$. Since Ca^{2+} and Mg^{2+} behave exactly the same, this convention is convenient shorthand.

Hardness (ppm $CaCO_3$)	**Classification**
< 15 ppm	Very Soft
15 ppm - 50 ppm	Soft
50 ppm - 100 ppm	Medium hard
100 ppm - 200 ppm	Hard
> 200 ppm	Very hard

The hardness of a sample of water can be measured by determining the concentration of the dissolved Ca^{2+} and Mg^{2+} ions. The procedure you will use is called a ***titration*** (See your text, pp. 153-155, for descriptions of titrations.)

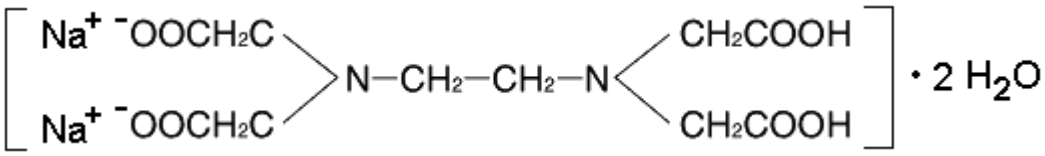
To analyze for Ca^{2+} and Mg^{2+} ions you will add a substance, Na_2EDTA, which will react with the metal ions and remove them from solution. You will know when you have added enough EDTA by using an indicator in the solution; the indicator will change color when all of the Ca^{2+} and Mg^{2+} ions have reacted.
The reactions of the metal ions are:

$$\mathbf{Ca^{2+} + EDTA^{2-} \rightarrow CaEDTA}$$

$$\mathbf{Mg^{+} + EDTA^{2-} \rightarrow MgEDTA}$$

Na_2EDTA is a complex molecule. Its name stands for ethylenediaminetetraacetic acid - disodium salt. The formula of disodium EDTA is $Na_2C_{10}H_{14}N_2O_8$. Even though its name and structure are complex, you only need to know that:

- **EDTA reacts with Ca^{2+} and Mg^{2+} in a one to one mole ratio**
- **The formula mass of Na_2EDTA dihydrate is 372.24 g/mole**



Disodium EDTA dihydrate (372.24 g/mol)

You will need to know the formula mass because you will be preparing a solution of EDTA with a specified molarity. By recording the exact volume of this solution that is needed to react with the Ca^{2+} and Mg^{2+}, the concentration of these ions can be determined.

The strategy for this lab will be as follows:

1. **Preparation of 0.010 EDTA Solution**: You will prepare a solution of EDTA with a concentration that is approximately 0.010 M. Because Na_2EDTA comes as a dihydrate that is ***efflorescent*** (efflorescent substances lose water of crystallization to the air), you cannot reliably weigh out the desired number of grams. The salt may be fully, or only partially, hydrated at the time you weigh it. The result is that you cannot be certain that you have prepared a 0.010 M solution. To determine the exact concentration of the solution you will need to standardize your solution (i.e. determine its molarity precisely) in the manner described in the procedure.
2. **Standardization of EDTA Solution**: The lab technician will have prepared a ***standard solution*** that contains exactly ***0.01000 M Ca^{2+}***. You will use the Ca^{2+} solution to "standardize" your EDTA solution. In other words, you will use the Ca^{2+} solution to determine the exact concentration of your EDTA solution by titrating the solution. You should find that the concentration of your solution is *very close* to 0.01000 M.
3. **Determine the Ca^{2+} Concentration in an Unknown and in Tap Water**: Once the EDTA solution has been standardized, you will use it to determine the hardness of two samples of water. One will be an unknown that the instructor will provide. The other will be a sample of water that you will bring from home. The unknown analysis must come within 5% of the true value for full credit.

Procedure

In this lab you will work individually. Begin by obtaining the following from the lab cart:

- A buret
- A stir plate and stir bar
- One 500 mL volumetric flask
- An unknown (record its number in lab your book and report the number to your instructor)

Complete the following steps:

1. Use the ***500 mL volumetric flask*** to prepare a 0.01000 M solution of Na_2EDTA, as described by your instructor.
2. Clean the buret carefully and rinse it twice with small amounts (5-10 mL) of your EDTA solution. Mount the buret on a ring stand using buret clamps. Use a funnel to fill the buret with your EDTA solution nearly to the top mark (i.e. Somewhere between the 0 and 5 mL marks—there's no need to waste time filling the buret to the top mark).
3. Record the exact reading of the liquid level in the buret to the nearest 0.01 mL. **Note:** *The burets can be read to the nearest 0.01 mL—be sure that all of your readings are accurate and consisten*t. Furthermore, be certain that you are reading the buret correctly. Check with your instructor if you are unclear.
4. Pipette 20.00 mL of the standard Ca^{2+} solution (i.e. the 0.01000 M Ca^{2+}) into a 125 mL Erlenmeyer flask. Add about 25 mL of deionized water.
5. Add a 3 or 4 drops of ***indicator*** and one dropperful of the ***buffer solution*** to the solution in the Erlenmeyer flask.
6. Place the stir bar in the flask, place the flask on the stir plate, and set the control for *gentle stirring*.
7. Slowly add your EDTA solution from the buret until the endpoint is reached. The ***endpoint*** is the point at which the solution in the Erlenmeyer flask turns a ***distinct blue***. As you near the endpoint, add EDTA drop by drop to avoid "overshooting the endpoint." Record to the nearest 0.01 mL the exact liquid level of EDTA in the buret, and calculate the volume used in the titration by subtracting the initial volume from the final volume of EDTA.
8. Repeat steps 3-7 two more times, using a fresh sample of standard Ca^{2+} solution each time. If the three trials do not agree to within 2% of each other (i.e. +/- 0.1 – 0.4 mL), do a fourth trial.
9. Calculate the exact concentration of your EDTA solution in moles/Liter from the data recorded.
10. Repeat steps 3-7 using your assigned unknown. Three trials are recommended, however, it is up to you to decide how many trials are necessary to ensure reliable results.
11. Repeat steps 3-7 using the sample of water from your home. If time permits and if you have sufficient EDTA solution, you may perform more than one trial. However, only one trial is required. **Take Note!!** The concentration of calcium and magnesium ions in the water from home is much lower than in the unknown. Hence, you will want to use a significantly larger volume of water (you decide how much) than for the unknown.

Analysis and Calculations

Be sure to do the following as part of your lab write-up:

1. Calculate the molarity of your EDTA solution, using the known concentration of the standard Ca^{+2} solution (i.e. 0.01000 M Ca^{2+}), the volume of the standard Ca^{2+} solution you used, and the volume of your EDTA solution. Average the results from your three trials, assuming they agree within +/- 2% of each other.
2. Calculate the molarity of your unknown using the concentration of your EDTA solution, calculated above, and the amounts of solutions used.
3. Do the same for your sample of water from home.
4. Convert the concentration of Ca^{2+}/Mg^{2+} ions to the standard reporting units of ppm $CaCO_3$. (See the next page for the method to do this.) Once you have completed the calculations, report your results to your instructor. Remember that you will receive a separate grade based on the accuracy of your results.

How to Convert from Molarity to Concentration in Parts per Million (ppm)

Parts per million, ppm, is a unit of concentration used for very dilute solutions. The units for ppm are grams solute per million grams solution. All masses below are in grams.

$$\text{ppm} = \text{mass fraction} \times 10^6$$

or

$$\text{ppm} = \left[\frac{\text{mass}_{\text{solute}}}{\text{mass}_{\text{solution}}}\right] \times 10^6$$

or

$$\text{ppm} = \left[\frac{\text{mass}_{\text{solute}}}{\text{mass}_{\text{solute}} + \text{mass}_{\text{solvent}}}\right] \times 10^6$$

Sample Problem. Calculate the concentration in ppm for 0.0100 M NaCl.

For one liter of 0.0100 M NaCl:

mass of solute = (0.0100 mol NaCl)(58.44 g NaCl/1 mol NaCl) = **0.5844 g NaCl**
mass of solution ≈ 1000 grams

Since the solution is very dilute: density of the solution ≈ d_{H20} = 1.00 g/mL
Hence, 1 liter of 0.0100 M NaCl = 1000 mL ≈ 1000 grams

$$\text{ppm} = \left[\frac{\text{mass}_{\text{solute}}}{\text{mass}_{\text{solution}}}\right] \times 10^6$$

$$\text{ppm} = \left[\frac{0.5844\ \text{g NaCl}}{1000\ \text{g}}\right] \times 10^6 = 588.4 \text{ ppm NaCl} = \boxed{\textbf{588 ppm NaCl}} \text{ (to 3 sig figs)}$$

How to use Volumetric Pipettes and Burets

In addition to reading the guidelines below, the following three instructional videos located at lab section of the Chem 161 website should be viewed:

1. How to Use a Volumetric Pipette
2. How to Set up a Burette
3. How to Perform a Chemical Titration

Volumetric Pipettes

1. Volumetric pipettes labeled TD (to deliver) at the upper end are designed to deliver the volume stated on the pipette. There will always be a small amount of liquid inside the tip after pipetting. If this liquid is blown out you will have delivered slightly more than the designed capacity of the pipette. Almost all of our pipettes are TD pipettes. However, other types do exist: Pipettes labeled TC (to contain) are designed to contain the volume stated on the pipette. Therefore, all liquid on a TC pipette should be expelled to obtain the desired volume.
2. If a drop of liquid hangs on the outside tip of the pipette the pipette tip should be dipped into the solution that has been pipetted to remove this drop. If this is not done you will have pipetted slightly *less* that the intended volume.
3. Volumetric pipettes, if clean and used properly, are precise to 0.01 mL. i.e. a 25 ml pipette delivers 25.00 mL, while a 20 mL pipette delivers 10.00 mL
4. **How to rinse a pipette**:
    - Fill about half full with D.I. water.
    - Rotate the pipette while holding it horizontally. Be sure that the water makes contact with all sides. Discard the water.
    - Repeat the previous two steps with the liquid to be pipetted.
    - Pipettes are clean if no liquid sticks to the sides after pipetting is completed. If sticking occurs the pipette must be returned for cleaning by the lab technician.
5. When finished, always rinse the pipette with D.I. at least two to three times before returning it.

Burets

1. Burets are designed to deliver any precisely measured volume of liquid up to the maximum of the buret capacity. Burets have scale divisions of 0.1 mL. Estimates between scale divisions can be made. Therefore, all volumes should be recorded to the nearest 0.01 mL (e.g. 22.48 mL, 20.00 mL, 15.60 mL, etc.)
2. **How to rinse a buret**:
    - Rinse the buret two to three times with about 15-10 rnL of D.I. water. Be sure that all inner sides make contact with the water. Run some of the water through the tip into a beaker to rinse the tip and stopcock. Discard the rinse water.
    - Rinse the buret twice with about 5-10 mL of the solution to be titrated. Be sure that all inner sides make contact with this solution. Run some of the water through the tip into a beaker to rinse the tip and stopcock. Discard the rinse solution into the proper waste container.
    - A buret is clean if no liquid sticks to the sides after releasing liquid. If sticking occurs the buret must be returned for cleaning by the lab technician.
3. **How to fill a buret:**
    - Use a clean and dry funnel to fill a rinsed buret to about the 0.00 mL mark with the solution to be titrated.
    - To expel air from the tip and stopcock, run some of the solution out into a small beaker. Tap the tip to dislodge air while letting the solution out. If the beaker is not clean and dry discard this solution in the proper waste container.
    - Fill the buret somewhere between the 0.00 and 5.00 mL marks. Record this volume.
    - Do not leave the funnel in the buret since liquid can drip down to give an inaccurate volume.
    - Now the buret is ready for use.
4. When finished, always rinse the buret with Dl. at least two to three times before returning it.

Lab Report Guidelines

As indicated previously, be sure to include all your lab data and calculations in your report, according to the guidelines in the "*Lab Notebook Policy*" handout. Below is a checklist of what should appear in each of the 5 sections of your lab report.

Lab 7 Report Checklist

Introduction

- Are the goals of the lab is stated clearly?
- Summary of background information concerning
  - Hard water
  - Why need to standardize EDTA
- Chemical equation for the reaction between Ca^{2+} and EDTA

Materials and Methods

- Procedure is brief, but detailed enough that a competent student could use it to replicate the experiment?
- Uses own words—doesn't plagiarize the procedure from the handout?

Results

Has a neat and orderly data ***ruled*** tables for the

- Standardization of EDTA: table includes...
  - Initial and final volumes of EDTA
  - Volume of standard Ca^{2+} solution (20.00mL)
  - Calculated value for the molarity of EDTA
- Analysis of the unknown: table includes...
  - Initial and final volumes of EDTA
  - Volume of the unknown solution (20.00mL)
  - Calculated value for the concentration of Ca^{2+} in the unknown in mol/L and ppm
- Analysis of tap water (optional)
- Correct use of sig figs → All volumes measured to 0.01 mL?

Analysis of Results

- Calculation of the molarity of your EDTA solution, using the known concentration of the standard Ca^{2+} solution (i.e. 0.01000 M Ca^{2+}), the volume of the standard Ca^{2+} solution (20.00 mL), and the volume of your EDTA solution.
- Calculation of the Ca^{2+} concentration in the unknown in mol/L and ppm using the molarity of EDTA calculated, above.
- Calculation of the Ca^{2+} concentration in tap water in mol/L and ppm $CaCO_3$ using the molarity of EDTA calculated, above.

Conclusion

- Uses "*bullets"* to state concisely the major conclusions:
  - Molarity of the EDTA
  - Ca^{2+} concentration in the unknown in mol/L and ppm $CaCO_3$
  - Ca^{2+} concentration in tap water in mol/L and ppm $CaCO_3$
- Summarizes major obstacles and sources of error?

Lab 7 Prelab Questions
Analysis of Hard Water

Name _____

Team No. _____ **Date** _____ **Section** _____

Instructions: Complete the following questions and hand in at the start of your lab period or when instructed by your instructor. Show your work with units and correct significant figures for all questions that involve a calculation. ***Circle numerical answers***.

1. A student prepared a solution of EDTA by placing 3.000g Na_2EDTA dihydrate in a 500.0 mL volumetric flask, dissolved the salt in D.I. water and then filled to the mark with D.I. water.
   a. Calculate the approximate molarity of the EDTA solution.

   b. Knowing that EDTA is an efflorescent substance, would you expect the actual molarity to be higher or lower than that calculated in part a? Explain.

2. a. A titration was performed to standardize an EDTA solution. Use the following data to calculate the molarity of the EDTA solution: 20.00 mL of a standard solution containing 0.01000 M Ca^{2+} required 24.33 mL EDTA to reach a distinct blue endpoint.

   b. Suppose the student performing the standardization in part a, above, washed his/her buret as per step 2 of the procedure, but neglected to rinse the buret with EDTA before titrating. How will this oversight impact the calculated molarity of the EDTA? Circle one of the following then explain your reasoning below. The calculated molarity will be
   a) artificially low. b) artificially high. c) unaffected by the oversight.
   Explanation:

   c. Suppose the student performing the standardization titration squirted some D.I. water into the flask containing the 20.00 mL of the standard Ca^{2+} solution. How will the addition of D.I. water impact the calculated molarity of the EDTA? Circle one of the following then explain your reasoning below. The calculated molarity will be
   a) artificially low. b) artificially high. c) unaffected by the oversight.
   Explanation: