

Experiment 8 - * Measurement of Water Hardness with EDTA Titrations

INTRODUCTION

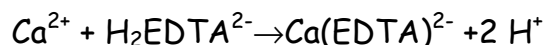
Water from lakes, rivers and wells often contains a significant amount of calcium and magnesium ions. These ions are what cause **hard water**. One characteristic of hard water is that it forms a precipitate with ordinary soap. This leads to the soap scum in a bathtub. The presence of these ions can also damage water heaters. Water hardness is reported as **mg calcium carbonate per liter of water**. (mg/L)

Calcium carbonate is an insoluble white precipitate. Reporting water hardness in this manner does not mean that the calcium carbonate is present in the water; instead it signifies how much calcium (and magnesium) carbonate would be present if carbonate was added to the solution. The table below shows the how water is classified on the hardness scale.

Classification	Concentration
Very hard	Over 300 mg/L calcium carbonate
Hard	150 to 300 mg/L calcium carbonate
Moderate	50 to 150 mg/L calcium carbonate
Soft	0 to 50 mg/L calcium carbonate

GOAL

The goal of this lab is to analyze water samples for their "total hardness", that is the combined concentration of Mg^{2+} and Ca^{2+} . This is a **titration experiment**: that is, you will be adding another compound (ethylenediaminetetraacetate, or **EDTA**) to the water. This compound reacts with the Ca^{2+} or Mg^{2+} ions as follows:



In this experiment, we will first use an EDTA solution to titrate standard calcium solutions and then use the same EDTA solution to titrate an unknown water sample and tap water. By comparison of the results, we will be able to calculate the hardness of the unknown sample and the sample of tap water.

BRIEF OVERVIEW

1. **EDTA** solution is added to a known amount of water until just enough is present to completely react with the calcium and magnesium ions.
2. In order for us know when **enough EDTA** has been **added**, a small amount of **indicator** (calmagite) is **added**.
3. In the **presence of metal ions**, the indicator is **red** in color.
4. In the **absence of metal ions**, the indicator is **deep blue** in color.

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When enough EDTA is added to react with all of the calcium and magnesium, the solution will turn **blue**.

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* Adapted from Laboratory Manual from Chemistry in Context 3rd Ed. (2000) W.J. Stratton, R.G. Silberman, C.L. Stanitskim A.T. Schwartz (eds.) McGraw-Hill Publishing

OUTLINE of EXPERMENTS

1. Do 4 titrations of water containing **known concentrations** of Ca^{2+} ions.
2. **Calculate the calibration factor (amount of EDTA for a known hardness)** for the EDTA solutions
3. Do 4 titrations for the water sample of **unknown hardness** and for **tap water**.
4. **Calculate the water hardness** of both the unknown and tap water.

****For simplicity, the amounts of Ca^{2+} and Mg^{2+} that are present in the water sample will be lumped together to determine the total hardness.****

EXPERIMENTAL PROCEDURE - Water hardness

1. Obtain a **24-well plastic well plate**.
2. Place the well plate on a piece of **white paper**.
3. Note the labels on each well of the well plate--you should use these to record the data.
4. **PRACTICE** using the **pipets** and **pipetors**
 - a. Fill a **pipet** with **deionized water**.
 - b. Try to **dispense drops** into the **well plate**.

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****This works best if you squeeze the bulb gently while the pipet is held vertically, directly over the center of a well. These pipets easily acquire air bubbles, which then lead to half drops, which will introduce error. If you have a bubble in the pipet that is about to be expelled, it is best to drop the next few drops into a waste well (or beaker) to get rid of the bubble****
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- c. You will also use **pipetors**.

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****Pipetors dispense exact volumes and will be set correctly for your experiment. The pipetors must have a blue plastic pipet tip on them when you use them. You will be shown how to use these before you begin the experiment.****
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5. Take **one test tube** that is clean and dry out of your drawer.
- 6 Fill the test tube about **1/3** of the way with the **EDTA solution**

Make sure you label the tube!!!

PRE-EXPERIMENT

Test the color change of the indicator with the **STANDARD SOLUTION**

- To **one well**, add **8-10 drops** of pure **deionized water**.(in the squirt bottles)
- Add **1 drop** of **pH 10 buffer** and **1 drop of indicator**. (the buffer and the indicator solutions (red solution) are located on the lab benches)

Stir the contents of the well with a clean stirring rod. The color should be **bright blue**. This is the color you should be looking for at the **end of the titration**.

EXPERIMENT#1

- Using a **pipetor**, (located with the standard solution on the **main** lab bench) dispense **exactly 1 ml** of the **standard solution** into **4 wells** in the well plates.
- To the **1st well**, add **1 drop of buffer plus 1 drop of indicator**.
- Start adding the **EDTA** solution (from your test tube) **one drop at a time**.

***** Stir after the addition of each drop and **KEEP TRACK of the # of DROPS!!!!*******

- Continue the **addition of the EDTA** until the **color starts to change** from **red** to **purple**.
- After each drop, wait a few seconds** to see if the color continues to change to **blue** or goes back to **red**.

The **endpoint** is reached when the solution becomes **pure blue**

- Record the number of drops** it takes to reach the **endpoint**.
- Repeat the procedure for the three other wells**.

- Record the data in the table below.

Standard solution

	Well 1	Well 2	Well 3	Well 4
# of drops				

Are your results consistent?

Calculate the average number of drops needed to reach the endpoint:

EXPERIMENT #2**UNKNOWN water sample and Tap Water**

- a. Using a **pipetor**, dispense **exactly 1 ml** of the **unknown solution** (located on the lab benches) into **4 wells** in the well plates.
 - b. To the **1st well**, add **1 drop of buffer plus 1 drop of indicator**.
 - c. Start adding the **EDTA solution one drop at a time**.
- ***** Stir after the addition of each drop and **KEEP TRACK of the # of DROPS!!!*******
- d. Continue the **addition of the EDTA** until the **color starts to change** from **red** to **purple**.
 - e. **After each drop, wait a few seconds** to see if the color continues to change to **blue** or goes back to **red**.

The **endpoint** is reached when the solution becomes **pure blue**
 - f. **Record the number of drops** it takes to reach the **endpoint**.
 - g. **Repeat the procedure for the three other wells**.
 - h. Record the data in the table below.

UNKNOWN Water Sample

	Well 1	Well 2	Well 3	Well 4
# of drops				

Are your results consistent?

Calculate the average number of drops needed to reach the endpoint:

Tap Water

	Well 1	Well 2	Well 3	Well 4
# of drops				

Are your results consistent?

Calculate the average number of drops needed to reach the endpoint:

CALCULATIONS

The hardness of the standard solution is 500 mg/liter (or 0.5 mg/ml). You can calculate the **calibration factor** (mg hardness/drop EDTA) for the EDTA solution as follows:

$$\frac{\text{mg hardness ref solution}}{1 \text{ ml ref solution}} \times \frac{1 \text{ ml ref solution}}{\text{ave drop EDTA}} = \frac{\text{mg hardness}}{1 \text{ drop of EDTA}}$$

1. Calculate the **mg hardness per drop of EDTA** for the **standard** solution:

This is a calibration factor that equates the mg of hardness to the drops of EDTA. You can then calculate the mg hardness in your unknown samples using the equation:

$$\frac{\text{drops of EDTA}}{1 \text{ ml solution}} \times \frac{\text{mg hardness}}{1 \text{ drop of EDTA}} = \frac{\text{mg hardness}}{1 \text{ ml sample}}$$

2. Calculate the **mg hardness per mL sample** for the **unknown sample**:

3. Calculate the **mg hardness per mL sample** for **tap water**:

4. Convert your values in #2 and #3 to **mg hardness per liter**.

5. Report the hardness of your samples in the table below. How hard is each sample of water? Refer to the table in the introduction to classify its hardness.

	Average # drops of EDTA used	Hardness (mg/ml)	Hardness (mg/L)	Hardness classification
Unknown Sample				
Tap Water				

6. In these titrations, there may be **uncertainty of one drop in identifying the end point**. If 20 drops of EDTA solution were used in a titration, what percentage uncertainty in the hardness is contributed by the one drop uncertainty (**therefore how does the hardness value for 20 drops compare with that for 21 drops**)? Would this change the hardness classification? Justify your answer with a calculation.