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Determination of Magnesium Using EDTA

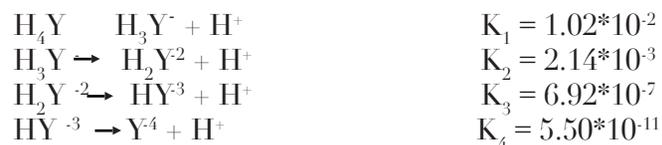
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Abstract

This laboratory determined the magnesium content in unknown 2, and hardness of water for UMKC's tap water, Parkville water, and deionized water. The concentration of EDTA was 0.01000M used to titrate unknown 2. The concentration of the magnesium solution was determined to be $1.730 \times 10^{-3} \text{M}$ with a standard deviation of 3.082×10^{-4} a standard error of the mean of $\pm 2.179 \times 10^{-4}$, and error limits of $\pm 9.371 \times 10^{-4}$ with 95% confidence. The hardness of UMKC tap water was 34.47mg/L, 20.20mg/L in Parkville, and 0.000 mg/L in deionized water.

The laboratory's purpose was to determine the magnesium content in unknown 2, and the hardness of water for the University of Missouri Kansas City's tap water, Parkville water, and deionized water. Hardness of water is determined by the presence of calcium ions and magnesium ions (1). According to NASQAN stations the predominant ion in Missouri is calcium (2). These metal ions come from dissolved minerals in aquifers and rivers. To measure the hardness of water requires a titration with a solution that will react in the same mole-to-mole ratio with both calcium ions and magnesium ions (2). The titration in this laboratory was done by using a standardized solution of ethylenediaminetetraacetic acid (EDTA) to titrate the four different solutions. EDTA was chosen as a titrant because it is a strong chelating agent. The word "chelate" is Greek for claw. The EDTA "clutches" the positive charges on metal ions. It has a unique chemical property that reacts with a large variety of metal

ions always in a one-EDTA (Y^{n-})-to-one metal ion (Me^{n+}) ratio. This forms a complex. EDTA has four hydrogens that can be lost (1).

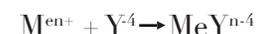
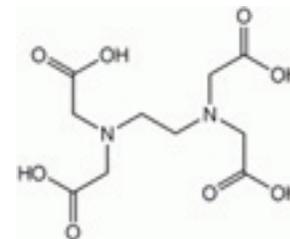


These four different oxidation states at different pH values. The EDTA used in this experiment was the hydrated form. The pH values control the relative concentration of H_4Y , H_3Y^- , H_2Y^{2-} , HY^{3-} , Y^{4-} . The molecular weight used in the calculations was the hydrated form of EDTA to account for the extra mass accumulated in a non-ideal environment. This decreased the possible margin of error, due to the presence of water, associated with partially hydrated forms.

The indicator used in this experiment was Eriochrome Black T. It was chosen because it also is a chelating agent. However, it has a lower affinity to the metal ions than EDTA. The weak bond is necessary in the indicator. If it had a strong affinity for the metal ions, then the EDTA would have to compete with the indicator. This would lead to error because there would be more ions present than the volume of the titrated EDTA would account for. Even though it weakly binds to metal ions and is easily kicked off by EDTA, only 2-3 drops of indicator were used to help reduce error.

Eriochrome Black T has one color when it is free in solution and a different color when it is bound to a metal ion and it can work at a variety of pH values. In an acidic solution the color change is from blue, as a free chelating agent, to orange when it is a ligand chelating agent. Between pH levels of 7-11, the color change is from red to blue. This change is more easily detected visually than red to orange. A basic pH was used in the experiment. EDTA can give up hydrogen ions that necessitate a buffer system to maintain a constant pH in the solution (3). The buffer used in this experiment was a mixture of ammonia and ammonium that had a pH of about 10.

Structure of EDTA



Materials and Methods

The glassware required for this experiment was 1000mL volumetric flask, three 250mL Erlenmeyer flasks, a 50ml burette, a 25mL, and a 50mL pipette. The chemicals needed were EDTA, the indicator Eriochrome Black T, the unknown 2 magnesium solution, an ammonia buffer solution, deionized water, UMKC tap water, and water from Parkville (3).

A 1000mL volumetric flask was cleaned with detergent and rinsed with deionized water. A 0.01M EDTA standard solution was prepared using hydrated EDTA. Approximately 3.7g were weighed on scale #4 in a weighing boat. This was washed with deionized water into the 1000mL volumetric flask. The original procedure called for a 250mL volumetric flask. It was changed so the solution had a smaller margin of error. The flask was filled half full with deionized water and swirled until the solid was dissolved. The solution was heated slightly for two minutes to aid in the dissolution of the EDTA. The flask was then filled to the calibration mark with deionized water and swirled (3).

Approximately 100mL of the unknown Mg^{2+} solution number 2 was put in a clean dry beaker and taken to the lab workstation. Three 25mL samples of unknown 2 were put in three 250mL Erlenmeyer flasks using a volumetric pipette. Two milliliters of the ammonia buffer solution and 8 drops of the indicator Eriochrome Black T were added to each sample. The solution became a deep magenta-red. The diluted EDTA solution was loaded into the burette and used to titrate the unknown solution until a blue endpoint (3).

A clean beaker was used to collect UMKC tap water. Triplicate samples of tap water were prepared. A 50mL pipette transferred the tap water to 250mL flasks to be tested for hardness. The buffer solution and indicator were added to the three samples. The solutions were titrated with EDTA until the blue endpoint was reached. The above steps were repeated with water from Parkville (3).

Deionized water was prepared as above with the buffer solution. However, when the indicator was added the solution immediately turned blue. No titration was necessary to reach the blue endpoint.

Results

The results showed the concentration of EDTA was 0.01000M that was used to titrate unknown 2. The concentration of the magnesium solution was determined to be 1.730×10^{-3} M. The hardness of tap water was 34.47mg/L, in Parkville water was 20.20mg/L and 0.000mg/L in deionized water. The standard deviation calculation $S = \sqrt{(\sum(x_i - \bar{x})^2 / n - 1)}$ produced a good standard deviation value, which means there is a large deal of accuracy to the measurement. The standard error of the mean $S_m = S / \sqrt{N}$ was also precise. The range for the 95% confidence level $\mu = \bar{x} \pm ts$ was good as well.

The parts per million calculations are milligrams per liter in metric units. The moles per liter were multiplied by the molecular weight. This yields grams per liter. The g/L are then multiplied by 1000 to give milligrams per liter. The ratio of mg/L and parts per million (ppm) is one to one.

$$\begin{aligned} \text{Mol/L} * \text{g/mol} &= \text{g/L} \\ \text{g/L} * 1000 &= \text{mg/L} \end{aligned}$$

Experimental Data: Hardness of Water

Scale #2

<u>Titration Data Mg²⁺</u>	<u>Trial 1</u>	<u>Trial 2</u>	<u>Trial 3</u>
Volume of unknown 2	25.00mL	25.00mL	25.00mL
Volume titrated	4.40mL	4.30mL	4.25mL
Concentration of Mg ²⁺	1.760×10^{-3}	1.720×10^{-3}	1.700×10^{-3}
Mean Concentration of Mg ²⁺		1.730×10^{-3}	
Mass of EDTA		3.7225g	
Concentration of EDTA		0.01000M	
Standard deviation [Mg ²⁺]		$\pm 3.082 \times 10^{-4}$	
Standard Error of the Mean		$\pm 2.179 \times 10^{-4}$	
Error Limits (95% CL)		$\pm 9.371 \times 10^{-4}$	
<u>Titration Data Tap Water</u>	<u>Trial 1</u>	<u>Trial 2</u>	<u>Trial 3</u>
Volume of Tap Water	50.00mL	50.00mL	50.00mL
Volume titrated	4.30mL	4.20mL	4.30mL
Concentration of ions	8.600×10^{-4} M	8.400×10^{-4} M	8.400×10^{-4} M
Mean Concentration of ions		8.500×10^{-4} M	
Hardness of water		34.47mg/L	
<u>Titration Data Parkville Water</u>	<u>Trial 1</u>	<u>Trial 2</u>	<u>Trial 3</u>
Volume of Parkville Water	50.00mL	50.00mL	50.00mL
Volume titrated	5.05mL	5.20mL	4.85mL
Concentration of ions	1.010×10^{-3} M	1.040×10^{-3} M	9.700×10^{-4} M
Mean Concentration of ions		1.010×10^{-3} M	
Hardness of water		20.20mg/L	
<u>Titration Data Deionized Water</u>	<u>Trial 1</u>	<u>Trial 2</u>	<u>Trial 3</u>
Hardness of water	0.000mg/L	0.000mg/L	0.000mg/L

Calculations

Concentration of EDTA

$$\begin{aligned} \text{Mass of sample} / \text{Molecular Weight of EDTA} / \text{1L} &= \text{moles of EDTA} \\ 3.7225 / 372.240\text{g/mol} &= 0.01000\text{M} \end{aligned}$$

Concentration of ions

$$\begin{aligned} \text{Volume titrated} * \text{concentration of EDTA} &= \text{moles of EDTA} \\ \text{Moles of EDTA} &= \text{moles of metal ions} \\ \text{Moles of metal ions} / \text{volume} &= \text{concentration of ions} \\ 0.00440 \text{ L} * 0.01000\text{M EDTA} &= 0.004400 \text{ moles EDTA} = \text{moles of metal ions} \\ 0.004400\text{mol} / 0.02500 \text{ L} &= 0.001760 \text{ M} \end{aligned}$$

Standard Deviation

$$\begin{aligned} &(\text{sum of } (x_i - \bar{x})^2 / \text{number} - 1)^{1/2} \\ &((0.00176 - 0.00173)^2 + (0.00172 - 0.00173)^2 + (0.00170 - 0.00173)^2 / 3 - 1)^{1/2} \\ &= 0.00173 \pm 3.082 \times 10^{-4} \end{aligned}$$

Standard Error of the Mean

$$S_m = S / \sqrt{N-1}$$

$$\text{Concentration of Mg}^{2+} = \pm 3.082 \cdot 10^{-4} / \sqrt{3-1} = 0.00173 \pm 2.179 \cdot 10^{-4}$$

Error Limits

$$\mu = X \text{ average} \pm ts / \sqrt{N} \quad t = 4.30 \quad (95\% \text{ CL})$$

$$0.00173 \pm (4.30 * 3.082 \cdot 10^{-4}) / \sqrt{3-1} = 0.00173 \pm 9.371 \cdot 10^{-4}$$

Hardness of Water

$$\text{Number of moles per liter} * \text{MW} * 1000 = \text{mg/L}$$

$$0.00085\text{M} * 40.08\text{g/mol} = 34.47\text{mg/L}$$

Discussion

The unknown 2 had the highest concentration of metal ions of $1.730 \cdot 10^{-3}\text{M}$ compared to UMKC tap water, which was greater than the concentration found in Parkville water. The deionized water had an ion concentration of zero. This is not surprising because deionized water has had all of the ions removed. This includes magnesium and calcium ions that constitute water hardness.

Kansas City has some of the best water in the Nation. The water quality in Kansas City was ranked third out of 100 American cities tested in March 2007 (4). It regularly exceeds the EPA's regulation standards of 180 containments, and tests for over 300 in more than 300,000 tests annually (4). Hard water can potentially cause a problem of calcium buildup in pipes and faucets. However, soft water can increase the salt concentration in a person's diet causing problems for individuals with high blood pressure.

UMKC's water was 34.47mg/L , which is moderately soft. Parkville tap water was 20.20mg/L , which is considered soft. It was surprising that Parkville's water was soft. Parkville is only a mile from the water treatment plant and borders the Missouri River. I expected that Parkville's water would be hard because the city is on the bank of the Missouri River which runs through limestone bedrock, and is full of metal which dissolved to make hard water. The water was probably freshly treated therefore reducing the number of ions in the water.

The University's water was harder than Parkville's water.

It is possible that the water came from an aquifer, which could account for the differences observed in the two tap water samples if they were from different sources. An underground aquifer would have a higher concentration of metal ions than a surface body of water such as a river. Aquifers are large underground water reservoirs that formed during the last glacial recession. The largest in North America is the Ogallala Aquifer that covers the central United States including Colorado, Nebraska, Kansas, and Oklahoma (2). It is heavily relied upon for irrigation of crops and as a municipal water source. Its volume has been decreasing rapidly because of the heavy usage.

It is likely that Kansas City uses the Missouri River for water instead of aquifers. The state line separates the use of aquifers versus the river. On the Kansas side of Kansas City the Ogallala aquifer is used and can be seen by the high concentration of metal ions in the water. On the Missouri side there is a steep drop in ion concentration indicating that the Missouri river is used (2). Kansas City sits at the bottom of the Missouri River basin, which has the water runoff of seven northern states. This provides easily accessible freshwater

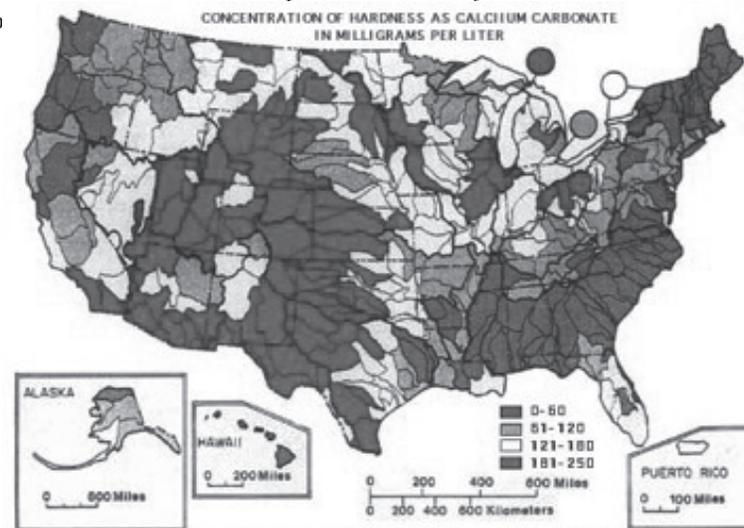


Figure 3.—Mean hardness as calcium carbonate at NASQAN stations during 1975 water year. Map at bottom is colored to show station data representing flow from the accounting unit.

<http://www.water-research.net/images/hardwatermap1.jpg>

Possible errors in this experiment included carbon dioxide dissolving in the water during the experiment, which would have made the solution softer. The indicator worked by binding as a chelating agent to the metal ions in the same way as EDTA. It was possible that not all the indicator was kicked off of the metal ions during titration. This would have made the concentration of metal ions lower than the true value. Titration error was another possibility.

The chemical used in the laboratory for testing the concentration of metal ions, EDTA, is a widely used chemical in the industrial world. When food is tinned or canned some of the metal from the container will dissolve into the food. This causes an unpleasant metallic taste. To avoid metal-flavored food while keeping an economic container, EDTA is employed to bind to metal ions so that they are not reactive, and thus not tasted. EDTA is not harmful when digested in small quantities and can be found in cans of Coke as well as much of the food that Americans eat. It is listed as an ingredient on the nutrition label of the foods that contain it. EDTA is also used as an alternative treatment to heavy metal poisoning of leads, cadmium, and zinc. It tightly binds to the metals in the bloodstream, becomes unreactive and is excreted out of the body (5).

References

- Determination of Magnesium Using EDTA. (2006). UMKC 341 Analytical Chemistry Handout Experiment 6.
- Hoekman, Dr. Theodore. *Heavy Metal Toxicology*. Retrieved April 15, 2007 From <http://www.hbci.com/~wenonah/hydro/heavmet.htm>.
- "Concentration of Hardness as Calcium Carbonate Milliliter." (2006). Retrieved November 15, 2006 From <http://www.water-research.net/images/hardwatermap1.jpg>.
- Pogge, Frank. (2006). Kansas City Missouri Receives and "A" In Men's Health Report Card. Water Services Department. Retrieved April 15, 2007 From <http://www.kcmo.org/water.nsf/web/home?opendocument>.
- Skoog; West; Holler; Crouch. (2004). *Fundamentals of Analytical Chemistry*: pp 235-600. Orlando, Florida: Harcourt College Publishers.