

DETERMINATION OF WATER HARDNESS BY EDTA TITRATION

Lab pH 5

From Gannon University SIM

INTRODUCTION

All natural waters contain dissolved cations and anions. Water dissolves many ions as it flows through minerals. Although water hardness is defined as the quantity of cations with a +2 or +3 charge, calcium ion and magnesium ion are the most common of such ions in natural water. The formation of solid calcium carbonate is an endothermic process. Thus, when water containing both carbonate and calcium ions is heated, calcium carbonate can precipitate out onto the walls of pipes, boilers, and household items such as tea pots. This can shorten the life-time of some of these items.

In addition, an insoluble scum develops when hard water comes into contact with soap. Both calcium and magnesium ions are responsible for this precipitate. This scum can be very difficult to clean.

However, there is some evidence that hard water has beneficial health effects. Selenium, for example, may help prevent cancer. Soft water drinking supplies have been associated with an increased heart attack risk.

The quantity of “hardness ions” will be determined by titration. EDTA, a weak acid, will be used as the titrant. In its ionized form, it is able to form soluble complexes with calcium and magnesium cations. The indicator added to the sample is Eriochrome Black T. Initially, the indicator will form a complex with the cations. When complexed it is red in color. As the EDTA is added dropwise to the sample, it replaces the Erio T and forms more stable complexes with calcium and magnesium. When the indicator is released by the metal ions, it has a distinct blue color. Therefore, the endpoint of the titration is marked by the color change from red to blue.

PURPOSE

The purpose of this experiment is to determine the hardness of water by measuring the concentrations of calcium and magnesium in water samples by titration.

MATERIALS

Eriochrome Black T indicator
pH 7 and 10 buffer
water samples
standardized 0.01M EDTA
buret, 25 ml and buret clamp
ring stand
DI H₂O

stir plate, bar & retriever
Kim Wipes
125mL Erlenmeyer flask
pH meter & electrode
25 mL pipet & pipet bulb
Beral Pipet
250mL beakers

SAFETY

- Always wear apron and goggles in lab

PROCEDURE

Total Hardness:

1. Pipet 25-ml of the water sample into an Erlenmeyer flask and dilute to a total volume of approximately 50 ml. Add at least one ml of pH 10 buffer solution (1/2 of a Beral pipet) to the sample. The pH should be 10. To check pH, standardize pH meter.
2. Standardize pH Meter:
 - a. Press **mode** to select pH.
 - b. Press **setup** twice and then **enter** to clear the existing standardization buffers.
 - c. Press **std** to access the Standardize screen. Immerse the electrode into pH buffer 7.0.
 - d. Press **std** again to initiate standardization. After the reading is stable, the meter will return to the measure screen. Remove electrode from buffer. Rinse off with DI water and blot dry with Kimwipe.
 - e. Repeat steps b and c with buffer 10.0.

3. Place the pH meter electrode into the Erlenmeyer flask. Make sure the meter is in **pH mode**. When the meter senses that the reading has stabilized, the stable icon will appear under the reading. Record the initial pH on the data sheet.
4. Remove the pH electrode from the flask. Rinse the electrode several times over the 250mL beaker, noting the pH reading on the pH meter. When pH hovers below 8.0, dry electrode with a Kimwipe and place in pH buffer 7.0.
5. Place the magnetic stirrer in the beaker and turn on the stirrer slowly, making sure that the bar does not hit the electrode.
6. Add a few drops Eriochrome Black T indicator to the Erlenmeyer.
7. Fill the buret with standardized EDTA. Record the initial buret reading.
8. Immediately begin to titrate the sample two drops at a time. Be careful to titrate slowly near the endpoint, as the color will take about 5 seconds to develop. Thus, add the last few drops at 3-5 second intervals. The endpoint color is blue.
9. Record the initial and final buret reading to the nearest 0.1 mL.

DETERMINATION OF WATER HARDNESS BY EDTA TITRATION

Name _____

Name _____

Period _____

Date _____

DATA

1. sample _____.
2. initial buret reading _____.
3. final buret reading _____.
4. total volume of titrant used _____.

CALCULATIONS

Hardness is expressed as parts per million (mg per liter) of equivalent CaCO_3 . For example, if the titration required 5 ml EDTA, the calculation would be:

$$\frac{5 \text{ ml } 0.01 \text{ M EDTA}}{0.025 \text{ L sample}} \times \frac{1 \text{ mg equivalent CaCO}_3}{1 \text{ ml of } 0.01 \text{ M EDTA}} = 200 \text{ ppm CaCO}_3$$

Calculate the hardness of your sample in ppm of calcium carbonate

Compare the hardness of your sample with others in your class.

1. Eriochrome Black T solution: Dissolve 0.5 g of dye in 100 g of triethanolamine or 2-methoxymethanol. Add 2 drops per 50 mL to be titrated.
2. EDTA solution: Dissolve 3.723 g of disodium ethylenediaminetetraacetate dihydrate in distilled water and dilute to 1.0 liter
3. Buffer solution: dissolve 16.9 g of ammonium chloride in 143 mL conc ammonium hydroxide. Add 1.25 g magnesium salt of EDTA and dilute to 250 mL with distilled water. Store up to one month.
4. **Alternative buffer:** Dissolve 1.179 g disodium salt of dthylenediaminetetraacetic acid dihydrate and 780 mg magnesium sulfate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$) or 644 mg magnesium chloride ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$) in 50 mL distilled water. Add this solution to 16.9 g NH_4Cl and 143 mL conc. ammonium hydroxide with mixing and dilute to 250 mL with distilled water.

Reference: Standard Methods for the Examination of Water and Wastewater, 20th ed., L. S. Clesceri, A. E. Greenberg, A. D. Eaton editors, 1998, American Public Health Association.

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pH

Water Hardness

02/08/05

For class of 7 students, 7 groups

- 24-125 Erlenmeyer flasks
- 24-50mL beakers
- 7-250mL beakers
- 8-pH meters and pH meter adapters (in pH meter box)
- 8- electrodes
- 7-ring stands
- 7-magnetic stirrer/hot plate
- 7- big stirring bars and 4 retrievers
- all-50 mL burets we have
- 7-buret clamps
- 7-buffer solution, 10
- 8- buffer solution, 7
- 8-wash bottles of distilled water
- box of-25mL pipets
- 7-green pipetters
- 7- red pipet bulbs
- 7-Kimwipes
- 8-empty 60mL plastic bottles for water hardness samples
- 7- masking tape and markers
- 4 Beral pipets labeled Eriochrome Black T indicator
- water hardness sample
- bottle of hardness buffer
- bottle of standardized 0.01M EDTA
- bottle of Eriochrome Black T indicator

- wash bottle of acetone, filled and capped with squirt stem attached