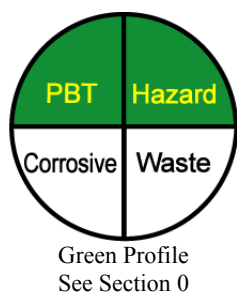


12. EDTA Titration of Ca^{2+} and Mg^{2+} in Natural Waters



The most common multivalent metal ions in natural waters are Ca^{2+} and Mg^{2+} . In this experiment, we will find the total concentration of metal ions that can react with EDTA, and we will assume that this equals the concentration of Ca^{2+} and Mg^{2+} . In a second experiment, Ca^{2+} is analyzed separately after precipitating $\text{Mg}(\text{OH})_2$ with strong base.

Reagents

EDTA: $\text{Na}_2\text{H}_2\text{EDTA} \cdot 2\text{H}_2\text{O}$ (FM 374.24), 0.6 g/student.

Buffer (pH 10): Add 142 mL of 28 wt% aqueous NH_3 to 17.5 g of NH_4Cl and dilute to 250 mL with distilled water.

Eriochrome black T indicator: Dissolve 0.2 g of the solid indicator in 15 mL of triethanolamine plus 5 mL of absolute ethanol. (Alternatively, Calmagite could be used by dissolving 0.05 g in 100 mL of water. The color changes are the same for both indicators.)

Hydroxynaphthol blue indicator: 0.5 g/student.

Unknowns: Collect water from streams or lakes or from the ocean. To minimize bacterial growth, plastic jugs should be filled to the top and tightly sealed. Refrigeration is recommended.

50 wt% NaOH: Dissolve 100 g of NaOH in 100 g of H_2O in a 250-mL plastic bottle. Store tightly capped. When you remove solution with a pipet, try not to disturb the solid Na_2CO_3 precipitate.

Procedure

1. Dry $\text{Na}_2\text{H}_2\text{EDTA} \cdot 2\text{H}_2\text{O}$ (FM 372.24) at 80°C for 1 h and cool in the desiccator. Accurately weigh out ~ 0.6 g and dissolve it ~~with heating in 400 mL~~ of water in a 500-mL volumetric flask. Cool to room temperature, dilute to the mark, and mix well.
2. Pipet a sample of unknown into a 250-mL flask. A 1.000-mL sample of seawater or a 50.00-mL sample of tap water is usually reasonable. If you use 1.000 mL of seawater, add 50 mL of distilled water. To each sample, add 3 mL of pH 10 buffer and 6 drops of Eriochrome black T indicator. Titrate with EDTA from a 50-mL buret and note when the color changes

from wine red to blue. Practice finding the end point several times by adding a little tap water and titrating with more EDTA. Save a solution at the end point to use as a color comparison for other titrations.

3. Repeat the titration with three samples to find an accurate value of the total $\text{Ca}^{2+} + \text{Mg}^{2+}$ concentration. Perform a blank titration with 50 mL of distilled water and subtract the value of the blank from each result.
4. For the determination of Ca^{2+} , pipet four samples of unknown into clean flasks (adding 50 mL of distilled water if you use 1.000 mL of seawater). Add 30 drops of 50 wt% NaOH to each solution and swirl for 2 min to precipitate $\text{Mg}(\text{OH})_2$ (which may not be visible). Add ~0.1 g of solid hydroxynaphthol blue to each flask. (This indicator is used because it remains blue at higher pH than does Eriochrome black T.) Titrate one sample rapidly to find the end point; practice finding it several times, if necessary.
5. Titrate the other three samples carefully. After reaching the blue end point, allow each sample to sit for 5 min with occasional swirling so that any $\text{Ca}(\text{OH})_2$ precipitate may redissolve. Then titrate back to the blue end point. (Repeat this procedure if the blue color turns to red upon standing.) Perform a blank titration with 50 mL of distilled water.
6. Calculate the total concentration of Ca^{2+} and Mg^{2+} , as well as the individual concentrations of each ion. Calculate the relative standard deviation of replicate titrations.