COMPLEXATION REACTIONS AND EDTA TITRATIONS

Background

For more details of the experiment, students must read the following **before coming to lab**. This information will also help with writing a detailed introduction for the lab report.

1. Yappert, M.C.; DuPré, D.B. *J. Chem. Educ.* "Complexometric Titrations: Competition of Complexing Agents in the Determination of Water Hardness with EDTA," **1997**, *74(12)*, 1422–1423.
2. Dahm, C.E.; Hall, J. W. *J. Chem. Educ.* "A Laser-Pointer-Based Spectrometer for Endpoint Detection of EDTA Titrations," **2004**, *81(12)*, 1787–1789.
3. Soroka, K.; Vithanage, R.S.; Phillops, D.A.; Walker, B.; Dasgupta, P.K. *Anal. Chem.* "Fluorescence Properties of Metal Complexes of 8-Hydroxyquinoline-5-sulfonic Acid and Chromatographic Applications," **1967**, *59*, 629–636.
4. Skoog, West, Holler, Crouch. "Chapter 17: Complexation and Precipitation Reactions and Titrations." *Fundamentals of Analytical Chemistry*.

Safety

Safety goggles and aprons must be worn in lab at all times. Store all waste in a correctly labeled waste container.

Procedures

Part A: EDTA Titrations of 0.01 M $MgCl_2$

1. Into one 150 mL beaker that contains a magnetic stir bar, pour 25 mL of 0.01 M $MgCl_2$ solution, 1 mL of NH_4OH buffer, and 10 drops of Calmagite. Put the beaker on a stir plate.
2. Turn the stir plate on and set it to a medium stirring speed (i.e. 3 – 5).
3. Clamp a burette, filled with 0.0100 M EDTA, above the beaker.
4. Plug the spectrometer into your computer and calibrate with DI water.
5. Use a plastic pipet to transfer about 3 mL of the solution in the beaker to a pre-rinsed cuvette (make sure light/detector is completely covered).
6. Place the cuvette in the spectrometer and measure the absorbance spectrum.
7. Carefully pour the solution in the cuvette back into the beaker and then use a pipet to transfer the remaining solution from the cuvette into the beaker.
8. Titrate slowly. Repeat step 5 – 7 after 10 mL and 20 mL of EDTA titrant is added. Then, titrate in 1 mL increments until the first color change happens. Record information about the color change.
9. Slowly add more titrant until the 2nd color change happens. *This is the end point.* Record the volume of EDTA at the end point. Take an absorbance spectrum.
10. Add another 5 mL of EDTA titrant. Repeat steps 5 – 7.

11. Repeat this titration one more time. When approaching the 2nd color change, carefully add EDTA titrant dropwise so that you can obtain a precise value for the volume. (When approaching the equivalence point, repeat steps 5 – 7 every 0.5 mL.)

Part B: EDTA Titrations of Unknown $MgCl_2$

The stockroom will provide a solution with an unknown concentration of $MgCl_2$. Repeat the procedures in Part A with this solution.

Part C: HQS Fluorescence

1. Prepare the solution for a fluorescence measurement. Use a micropipette to transfer a 500 μL aliquot of unknown solution from the hood to a flask with 25 mL of 1 mM HQS solution. Mix the solution thoroughly. Use a plastic pipet to transfer a few drops of the solution above onto pH paper. Record the color and pH of the solution.
2. Absorbance Spectrum
    a. Open *Logger Pro*. If the program does not automatically recognize the spectrometer, unplug it and plug it back in. Go to *Experiment* and choose *Calibrate*. Let the lamp warm up for at least 90 seconds. When the spectrometer is ready, fill a clean cuvette with water to use as a blank.
    b. Transfer 1.5 mL of the solution made in Step 1 in a pre-rinsed cuvette. Press the *Collect* button. Once the spectrum is acquired, press *Stop*. Rinse the cuvette. Save the spectrum. Examine the spectrum, and then chose the best excitation wavelength. Recall that the best wavelength is where the absorption is reasonably strong but the emission is weak. With only two excitation wavelengths at your disposal (405 nm and 500 nm), the choice should be quite obvious.
3. Fluorescence Spectrum
    a. Go to *Experiment* and choose *Calibrate*. Let the lamp warm up for at least 90 seconds. When the spectrometer is ready, fill a clean cuvette with 1 mM HQS solution to use as a blank.
    b. Go to *Experiment* and choose *Change Units*. Change the units to fluorescence, selecting the appropriate fluorescence wavelength.
    c. Transfer 1.5 mL of the solution made in Step 1 into a pre-rinsed cuvette. Press *Start*. Wait a few seconds and then record the wavelength of maximum fluorescence and the fluorescence intensity at that wavelength. Save the spectrum. Remove the cuvette, and rinse it well. Save all spectra, because the data is needed for your lab report.
    d. Plot the data below as fluorescence intensity versus concentration of $MgCl_2$. Does the unknown signal fall within the calibration range? If not dilute the unknown by an appropriate factor (e.g., by 2), and collect another spectrum.

e. Use the plot to determine the $MgCl_2$ concentration of the unknown solution.

Fluorescence Intensity	**Volume of 0.01 M $MgCl_2$ (mL)**	**Volume of 1 mM HQS (mL)**
0.031250902	0.8	100
0.049490754	0.8	50
0.059803507	2	100
0.085834029	3.6	100

Part D: Shutting Down

The solutions produced during this lab should be poured into the waste container located in a fume hood. Please turn off the program and disconnect the spectrometer from the computer (following proper steps for disconnecting USB powered hardware). Return kits to the stockroom with clean glassware.