COMPLEXOMETRIC TITRATION WITH EDTA

BACKGROUND

This is a two-part mini-project on the application of ethylenediaminetetraacetic acid (EDTA), a versatile titrant for the quantitative determination of metal ions. In the first part, the disodium salt of EDTA is standardized by a standard magnesium salt, thereby gaining a first-hand experience on the nature of complexometric titration. In the second part of the experiment, you will be assigned a commercial sample to determine the concentration of a metal ion by using the standard EDTA solution that you have prepared in the first part. It is your responsibility to discover the appropriate methodology from experiment and literature search.

Disodium dihydrogen ethylenediaminetetraacetate is available commercially of analytical reagent purity, and can be used as a primary standard. Tetrasodium salt of EDTA can also be used. To use the reagent grade EDTA as the primary standard one must dry the sample at 80 °C to expel the superficial moisture. The reagent has an exact formula Na₂H₂C₁₀H₁₄O₁₀N₂2H₂O (372.25 amu). Alternatively, one can use tetrasodium EDTA: C₁₀H₁₆N₂Na₄O₈·2H₂O (416.21 amu).

With EDTA magnesium ion forms a soluble, 1:1 complex that is stable enough to permit a titration. However, the stability of the complex is too low to require a high pH value in order to shift the equilibrium

\[ \text{Mg}^{2+} + \text{H}_2\text{Y}^2- \rightarrow \text{MgY}^2- + 2\text{H}^+ \]

toward the right and thereby assure virtually complete complexation of the magnesium ion. This is achieved by buffering the solution to pH 10. The complex of magnesium ion with the indicator, Erichrome Black T (EBT) in solution is a brilliant wine red. The reaction of this indicator complex with EDTA is somewhat slow at room temperature; consequently, warming of the solution is recommended. The end point corresponds to the disappearance of the last trace of a red tint to leave a pure blue.

In the following procedure you will prepare a disodium EDTA solution and standardize the solution by titrating it with a standard magnesium solution.

PROCEDURE

PART I: Preparation of solutions

Preparation of 0.01 M EDTA Solution: Dissolve 3.723 g of disodium EDTA in water and dilute to 1 liter in a volumetric flask with distilled deionized water. Store the solution in a polyethylene bottle. If tetrasodium EDTA is used you should use appropriate amount to make 0.01 M EDTA.

Preparation of Mg(II) Solution: Dissolve 0.061 g, precisely weighed, of pure magnesium turnings in a minimum volume of 6 M hydrochloric acid (start with ~10mL, do not exceed 20mL). Nearly neutralize (neutral to litmus pH paper) the solution with 1 M sodium hydroxide. This step must be done very carefully so that Mg does not precipitate as Mg(OH)₂(s). Dilute to the mark in a 250.0 mL volumetric flask. Calculate the exact molarity of Mg(II) in the solution.
**Preparation of Buffer Solution:** Add 142 mL of concentrated ammonia solution to 17.5 g of ammonium chloride, and dilute to 250 mL with distilled water. Calculate the pH of the buffer solution. *(Groups will not be making this buffer because there is not enough concentrated ammonia in the lab; likely an ammonia buffer will be made in advance for the entire class).*

**PART II. Titration of EDTA**
Dilute 25.00 mL of the Mg(II) solution to 50.0 mL, add 2.0 mL of the buffer solution (pH 10) and 3-4 drops of Erichrome Black T (EBT) indicator. Titrate with 0.01 M EDTA until the color changes from red to pure blue. The last trace of a reddish shade should disappear at the end point. Complex formation does not take place immediately; titration should therefore be conducted slowly near the end point. Alternatively, the solution may be warmed to enhance the reaction. Repeat the titration three times and calculate the exact molarity of EDTA solution (RSD<0.5%), the following relation might prove useful:

\[ 1 \text{ mL of 0.01 M EDTA} = 0.2432 \text{ mg of Mg} \]

**NOTES**
Preparation of EBT Indicator: Dissolve 0.2 g of the dyestuff in 15 mL of triethanolamine and 5 mL of absolute ethanol.
COMPLEXOMETRIC TITRATION OF ZINC IN MOUTHWASH USING STANDARD EDTA SOLUTION

METHOD

The objective of the EDTA complexometric titration is the analysis of Zn content in commercial mouthwash. This is a visual titration, and the mouthwash itself is a colored (light red or blue) aqueous solution, also containing numerous organic compounds. Therefore, the first critical step is the preparation of the mouthwash sample through oxidation of organic compounds, most importantly, to eliminate the color of the solution. You can then perform the EDTA titration. You have to discover the detail methodology for this experiment. Use the following procedure only as your guide. (Remember to note all observations and volumes used.)

1. Pipet 10.0 mL aliquot of mouthwash into 250 mL Erlenmeyer flask. Dilute the sample with 20 mL water.
2. Add concentrated nitric acid dropwise with heating and constant mixing to oxidize sample to a faintest yellow color. Note the number of drops added. This procedure must be done in the hood.
3. Slowly add 10-15 drops of 30% hydrogen peroxide in the hot solution to complete the oxidation. Note the solution color.
4. After oxidation, dilute the solution to about 50 mL with distilled water.
5. Neutralize the acidic solution to litmus blue very slowly with NaOH solution.
6. Add 2-3 mL of pH 10 ammoniacal buffer solutions.
7. Add 6 drops of Erichrome black T (enough to impart a noticeable color change toward red).
8. Titrate with your standardized EDTA until blue end point, making sure EDTA volume is within 20-30 mL. You may have to dilute your original EDTA titrant to obtain this value.
9. Calculate the average wt% (g/100 mL) and relative standard deviation of Zn in the mouthwash.

QUESTIONS
1. Explain with equations how the EBT changes its color during the titration.
2. Explain the role of ammoniacal buffer in the titration Zn with EDTA.