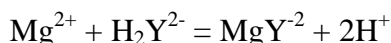


## Determination of Mg by Titration with EDTA

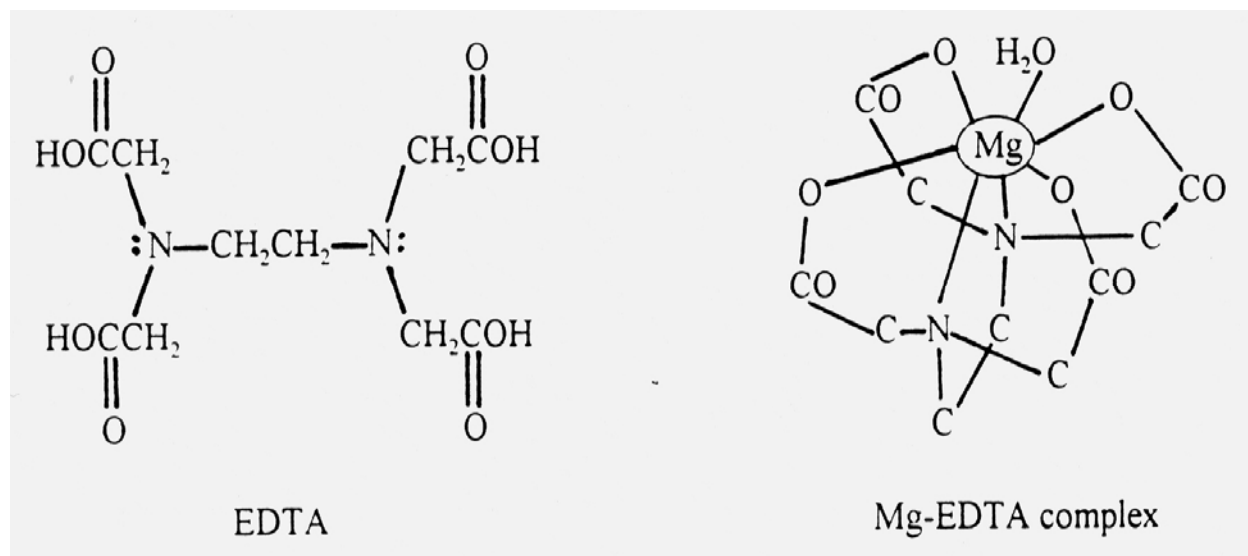
### INTRODUCTION:

Many metal ions react with electron pair donors to form coordination compounds or complex ions. The formation of a particular class of coordination compounds, called chelates, are especially well suited for quantitative methods. A chelate is formed when a metal ion coordinates with two (or more) donor groups of a single ligand. Tertiary amine compounds such as ethylenediaminetetraacetic acid (EDTA) are widely used for the formation of chelates. Complexometric titrations with EDTA have been reported for the analysis of nearly all metal ions.

Because EDTA has four acidic protons, the formation of metal-ion/EDTA complexes is dependent upon the pH. For the titration of  $\text{Mg}^{2+}$ , one must buffer the solution to a pH of 10 so that complex formation will be quantitative. The reaction of  $\text{Mg}^{2+}$  with EDTA may be expressed as:



The structure of EDTA and the magnesium-EDTA complex (without the hydrogen atoms) is shown below:



The endpoint of the titration is determined by the addition of Eriochrome Black T, which forms a colored chelate with  $\text{Mg}^{2+}$  and undergoes a color change when the  $\text{Mg}^{2+}$  is released to form a chelate with EDTA. While it is possible to achieve relatively good results by titration with EDTA prepared directly from the solid, better results should be obtained when the EDTA is standardized against a solution containing a known amount of metal ion. You will be provided with a standard solution of  $\text{Zn}^{2+}$  which you will use to standardize your EDTA solution.

## **REAGENTS AND APPARATUS**

*EDTA* ( $\text{Na}_2\text{H}_2\text{Y} \cdot 2\text{H}_2\text{O}$ )

*pH 10 buffer* (2000 mL has been prepared by dissolving 140.0 g of  $\text{NH}_4\text{Cl}$  in 650 mL of deionized water, adding 1136 mL of conc. ammonia and diluting to 2000 mL)

*Eriochrome Black T* (ground 1:10 with  $\text{NaCl}$ )

*pHydrion paper*

*Standard Zn solution:* (This has been prepared for you.) An accurate mass (~1.3g) of pure zinc has been dissolved in a small volume (~15 mL) of 6M  $\text{HCl}$ . The dissolved zinc was quantitatively transferred to a 2 L volumetric flask and diluted to the mark. This ~0.01 M (you need to find the exact concentration!!) solution serves to standardize the EDTA solution. Remember that the results from your magnesium determination depend on the accurate preparation of this solution.

## **PROCEDURE**

### **PART A -- PREPARATION OF STANDARD 0.01 F $\text{Na}_2\text{H}_2\text{EDTA}$ SOLUTION**

1. Weigh about 3.8 g of the disodium EDTA salt ( $\text{Na}_2\text{H}_2\text{Y} \cdot 2\text{H}_2\text{O}$ ) (*what balance should you use?*) into a 1 liter volumetric flask, dissolve and dilute to the mark with deionized water and mix well.

### **PART B – STANDARDIZATION OF THE EDTA SOLUTION**

1. Pipet exactly 25 mL of standard Zn solution into each of three Erlenmeyer flasks. Add 20 mL of pH 10 buffer (in the hood), 15 mL of water, stir, and add a few crystals of the Eriochrome Black T indicator. It is critical to add only enough indicator to produce a light wine-red color.
2. Titrate with your EDTA solution until the color changes from wine-red to a clear blue.
3. Use these results to determine the molar concentration of the EDTA solution for use in the titration of your unknown Mg solution.

### **PART C: DETERMINATION OF UNKNOWN MAGNESIUM**

1. Your unknown for this experiment is a solution. When you obtain your unknown, you need to quantitatively transfer it to a 100 mL volumetric flask and dilute it to the mark, resulting in the "prepared" unknown solution. You are to report the results of this "prepared" unknown.
2. Transfer exactly 10 mL of the prepared unknown solution into 3 or 4 Erlenmeyer flasks. Add approximately 15 mL of pH 10 buffer in the hood and 20 or 25 mL of distilled, deionized water to each flask.
3. Add a few crystals of Eriochrome Black T indicator -- it is crucial that you only add enough indicator to produce a light, wine-red color.
4. Titrate each solution with your standardized EDTA solution to a clear blue color.
5. Report your results as percent magnesium (% w/v) in your "prepared" unknown sample. Also report the 95% confidence interval.

## Information, Hints and Help for Mg Determination

### Preparation:

- To prepare the standard EDTA solution used in this experiment, you must first be aware that the structure of the EDTA listed in your text and lab manual is not the compound you will be weighing out to make the solution. The salt you will use is the disodium salt of EDTA. The formula weight can be obtained from the bottle.
- Since you are going to standardize the EDTA solution with a standard Zn solution, you need not dry the EDTA.
- After making your EDTA solution, if you wish to keep some for an additional lab period, store it in a plastic bottle, not a volumetric flask.
- The Zn solution used for standardization may be prepared for you in advance. Consult your class instructor for this information. Be sure to note the actual concentration of the Zn solution, as it will not be exactly 0.01 M.

### Technique:

- Eriochrome Black T exists as a wine-red complex when  $\text{Mg}^{+2}$  is present in solution at pH = 10. When the EDTA has chelated all the  $\text{Mg}^{+2}$  present in solution, the indicator (free and uncomplexed to  $\text{Mg}^{+2}$ ) will be robin's egg blue. This color change marks the endpoint. Add only very small quantities of the indicator are needed. Do your best to keep the intensity of the indicator color relatively weak and consistent from sample-to-sample.
- It is best to test the indicator color change before trying the titration. Try it either with tap water or a dilute  $\text{Mg}^{+2}$  solution. Using a sample with a small amount of  $\text{Mg}^{+2}$  and performing a low volume sample titration will help you understand the endpoint changes.
- A trick which makes the end point much easier to detect for some is to use methyl red indicator solution in addition to the Eriochrome Black T. This changes the two endpoint colors to orange initially and green at the endpoint. Be careful when doing this step though, as the additions must be done in a way as to keep the intensities of colors roughly the same in all samples.

### Calculations:

- The concentration of magnesium that you report is that of the unknown you received after it was diluted to 100 mL in the volumetric flask (the "prepared" unknown).