

Ministry Of Higher Education and scientific Research

Al-Mustansiriya University

Collage of Science

Department of Chemistry

Practice Volumetric Chemical Analysis

First year / 2012



Edited by

Ruba Fahmi Abbas

Supervised by

Lecturer Dr.

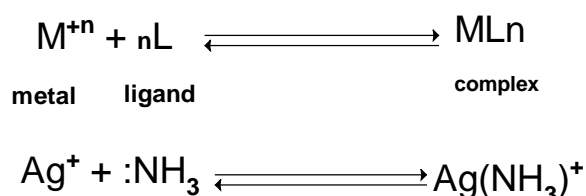
Mohammed Abdul-Hadi Khalil

Lecturer Dr.

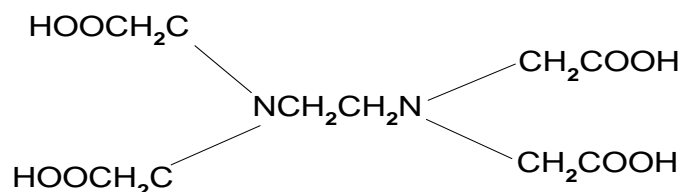
Abbas Shebeeb Hasan

4// Complex –formation Titration:-

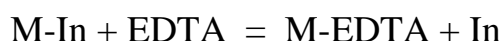
Most metal ions react with electron- pair donors to form coordination compounds or complex ion. The donor species or **ligand** must have at least one pair of unshared electrons available for bond formation. Water, ammonia, and halide ions are common inorganic ligands.



The **coordination number** of a cation is the number of covalent bonds that a cation tends to form with electron donor groups. Titrimetric methods based upon complex formation. A ligand that has more than two donor group chelating agents are known. Like EDTA (Ethylene di amine tetra acetic acid)



The use of a metal ion indicator in an EDTA titration may be written as:



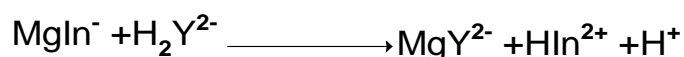
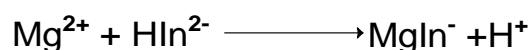
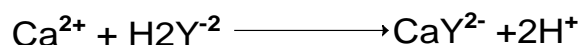
This reaction will proceed if the metal-indicator complex $M-In$ is less stable than the metal-EDTA complex $M-EDTA$. The former dissociates to a limited extent, and during the titration the free metal ions are progressively complexed by the EDTA until ultimately the metal is displaced from the complex $M-In$ to leave the free indicator (In).

The majority for EDTA titration is Eriochrome black T, This substance is sodium 1-(1- hydroxy-2-naphthylazo) -6-nitro-2-naphthol-4-sulphonate. In strongly acidic solutions the dye tends to polymerise to a red-brown product, and consequently the indicator is rarely applied in titrations of solutions more acidic than $pH = 6.5$.

Experiment No.(7):- Determination of water hardness

Theory:-Water hardness was defined in terms of the capacity of cation in the water to replace the sodium or potassium in soaps and form sparingly soluble products.

Water hardness determined by an EDTA titration after the sample has been buffered to pH 10. Mg, which forms the least stable EDTA complex of all of cation in water sample, is not titrated until enough reagent has been added to complex all of other cation in the sample. Therefore, Mg ion indicator such as EBT, can serve as indicator in water hardness titration. (Indicator EBT = H_3In^- , EDTA= H_4Y).



Red colorless colorless blue

Procedure:-

1. Dry 3 gm $Na_2EDTA \cdot 2H_2O$ ($Na_2H_2Y \cdot 2H_2O$) at $80\ C^0$ for 2 hour, cold in desiccator for 30 min.
2. Take 0.2 gm and transfer into 50 ml volumetric flask after dissolved in 40 ml D.W(de-ionized), stirring, and complete the volume to the mark.
3. Calculate the molarity of EDTA:-

$$M_{EDTA} = \frac{\text{Wt. of } Na_2H_2Y \cdot 2H_2O \text{ (gm)}}{\text{M.wt } Na_2H_2Y \cdot 2H_2O \text{ (g/mol)}} * \frac{1000}{50}$$

4. Titration: to 50 ml sample of water add 2 ml buffering solution ($NH_4Cl + NH_4OH$, pH=10) and 30-40 mg EBT indicator , titrate with standard 0.01 M EDTA until the color changes from red to blue, **should there be no Mg**

present in the sample of water it is necessary to add 0.1 ml Mg-EDTA solution (0.1 M) before adding indicator. The total hardness is expressed in parts of CaCO₃ per million of water. (note: if the water contains traces of interfering ions, then 4 ml of buffering solution should be added, followed by 30 mg hydroxyl ammonium chloride and then 50 mg A.R potassium cyanide before adding the indicator.

Calculation:- Calculate total hardness is expressed in parts of CaCO₃ per million of water:-

$$\text{ppm(mg/l)} = \frac{\text{Wt.(gm)}}{\text{V(ml)}} * 10^6 \implies \frac{\text{Wt.(gm)}}{\text{V(ml)}} = \frac{\text{ppm (mg/l)}}{10^6} \dots 1$$

$$(M * V)_{\text{CaCO}_3} = (M * V)_{\text{EDTA}}$$

$$\frac{\text{Wt.(gm)}}{\text{M.Wt}_{(\text{CaCO}_3)}} * \frac{1000}{\text{V (ml)}} * V_{(\text{sample})} = (M * V)_{\text{EDTA}} \dots 2$$

Put eq. (1) in eq.(2), we have:-

$$\frac{\text{ppm(mg/l)}}{10^3} * \frac{1}{\text{M.Wt}} * V_{(\text{sample})} = (M * V)_{\text{EDTA}}$$

$$\text{ppm(mg/l)} = \frac{(M * V)_{\text{EDTA}} * 10^3 * 109.09}{50 \text{ ml}}$$

Discussion:-

1. Why Mg-EDTA is added?
2. What is the de-ionized water?
3. What is the coordination number?
4. What is the EBT, EDTA, and ligand?
5. How many type of hardness and must be made?