Chemistry 203

Determination of EDTA in Shampoo and Magnesium in an Unknown

Purpose:

- To learn the principles and techniques of complexometric titration.
- To prepare a standard Magnesium solution.
- To standardize an EDTA solution using the standard Magnesium solution.
- To determine the % mass of EDTA in a shampoo solution.
- To determine the amount of Magnesium in an unknown solution.

Theoretical Background:

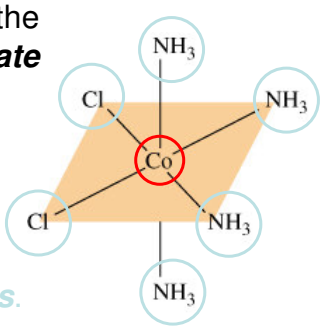
Coordination Compounds:

A ***coordination compound*** typically consists of a complex ion and a counter ion.

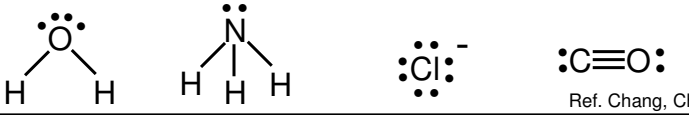
In a coordination compound, part of the molecular bonding is of the ***coordinate covalent*** type.

A ***complex ion*** contains a ***central*** metal cation bonded to one or more electron-donating groups (molecules or ions).

The molecules or ions that surround the metal in a complex ion are called ***ligands***.

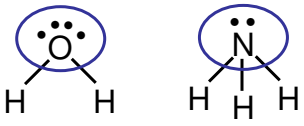


A ligand has **at least one** unshared pair of valence electrons



Ref. Chang, Chemistry, 8th ed., Fig. 22.3

The atom in a ligand that is bound directly to the metal atom is the ***donor atom***.

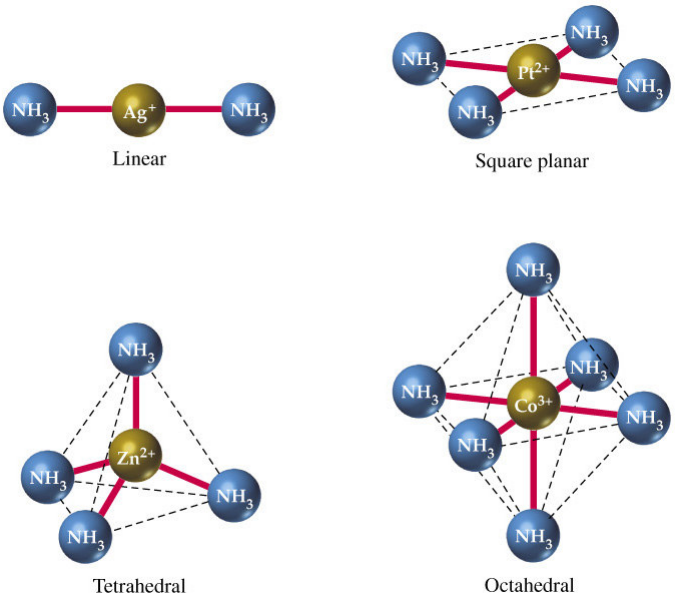


The number of donor atoms surrounding the central metal atom in a complex ion is the ***coordination number***.

A ***chelate*** is a cyclic complex formed when a cation is bonded by two or more donor groups contained in a single ligand.

Complexometric Titration: A technique of volumetric analysis in which the formation of a colored complex is used to indicate the end-point of a titration.

Common Structures of Coordination Compounds:

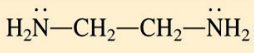


Ref. Hill and Petrucci, General Chemistry, 3rd ed., Fig. 20.11

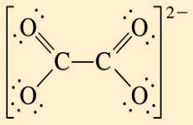
Types of ligands:

- Based on charge:
  - Neutral:
    - NH_3
    - $NH_2CH_2CH_2NH_2$
    - H_2O
  - Negatively Charged:
    - Cl^-
    - OH^-
    - F^-
    - CN^-
    - SCN^-
- Based on number of electron-donating groups (electron pairs):
  - One pair: Monodentate ligand
    - NH_3
  - More than one pair: Multidentate ligand
    - bidentate $NH_2CH_2CH_2NH_2$
    - tridentate
    - tetradendate
    - pentadentate
    - hexadentate EDTA
    - etc…

Ethylenediamine

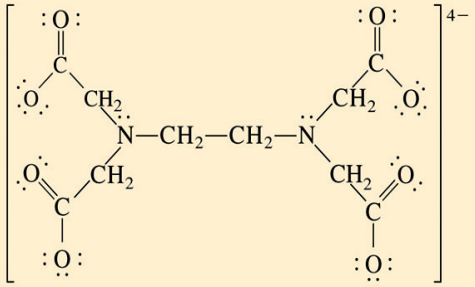


Oxalate ion



Polydentate ligand

Ethylenediaminetetraacetate ion (EDTA)

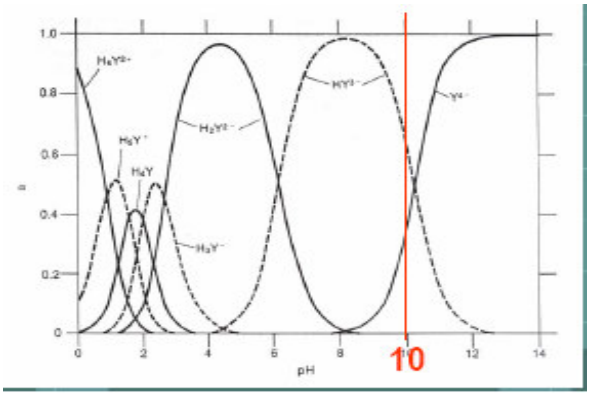


Ref. Chang, Chemistry, 8th ed., Table 22.3b

Forms of EDTA at different pH

- Six successive ionizations:

$H_6Y^{2+} \leftrightarrow H_5Y^{+} \leftrightarrow H_4Y \leftrightarrow H_3Y^{-} \leftrightarrow H_2Y^{2-} \leftrightarrow HY^{3-} \leftrightarrow Y^{4-}$



Use of EDTA

- EDTA is used in shampoos and detergents to act as a builder (chelates metals) especially as a replacement for phosphates, a major nutrient in wastewater.
- EDTA reduces calcium and magnesium hardness in water by binding to them, thus softening the water and allowing the surfactants to clean properly.

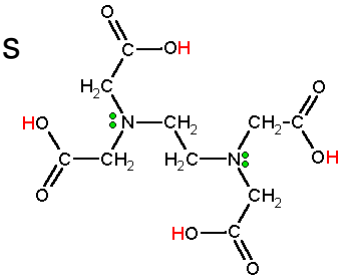
Environmental Problem of EDTA

- EDTA does not biodegrade easily.
- Once EDTA is introduced into the general environment, there is the potential of re-dissolving toxic heavy metals trapped in underwater sediments thus permitting them to re-enter and re-circulate in the food chain.

Complexation with EDTA

- Most complexometric titrations are carried out with EDTA, a hexadentate ligand.
- The reaction is stoichiometric and instantaneous:

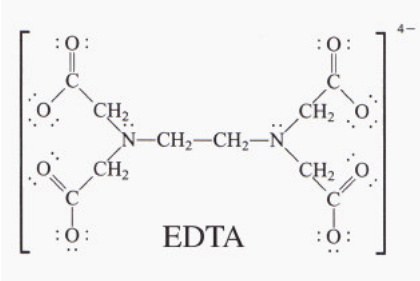
  $Mg^{2+} + Y^{4-} \rightarrow MgY^{2-}$



Ref. : www.chm.bris.ac.uk/ motm/edta/edta.htm

- Formation constant:

$$K_f = \frac{[MgY^{2-}]}{[Mg^{2+}][Y^{4-}]} = 4.9x10^8$$



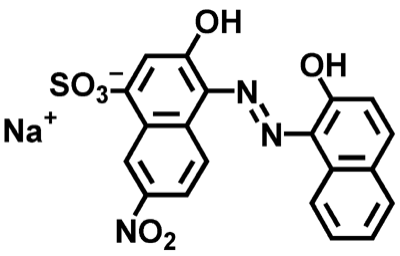
Direct Titration and Back Titration:

In a direct titration, analyte is titrated with standard EDTA. The analyte is buffered to a pH at which the formation constant for the metal-EDTA complex is large and the color of the free indicator is distinctly different from that of the metal-indicator complex.

In a back titration, a known excess of EDTA is added to the analyte. Excess EDTA is then titrated with a standard solution of a metal ion. A back titration is necessary if analyte precipitates in the absence of EDTA, if it reacts too slowly with EDTA, or if it blocks the indicator.

Indicator:

- Eriochrome Black T
  - It is a typical metal-ion indicator used in the titration of several cations.
  - It is a triprotic acid that have different colors of the acids and their conjugate bases. It can also behave as an acid-base indicator.
- $H_3E \leftrightarrow H_2E^- \leftrightarrow HE^{2-} \leftrightarrow E^{3-}$
- HE^{2-} is predominant at pH 10.0



http://www.gfschemicals.com/chemicals/gfschem-246.asp

Reaction in the presence of Erio-T

- Mg^{2+}, Y^{4-} and MgY^{2-} are all colorless.
- It is convenient to carry out the reaction at pH=10.0 (use an NH_3/NH_4Cl buffer).
- Erio-T complexes with Mg^{2+} at pH 10.0
- Until the equivalence point, the indicator complexes with the excess Magnesium:

$$Mg^{2+} + HE^{2-} \rightarrow MgE^- + H^+$$

blue — wine red

When EDTA becomes present in slight excess:

$$MgE^- + HY^{3-} \rightarrow MgY^{2-} + HE^{2-}$$

wine red — blue

Procedure:

- Preparation of standard $MgSO_4$ solution:
- Preparation of comparison solution:

  All end-point determinations will be made in comparison to this solution.
- Standardization of EDTA solution.
- Preparation of the shampoo solution.
- Determination of EDTA in shampoo via back titration.
- Determination of Mg in Unknown via direct titration.

Note:
Back Titration

In the case when the amount of EDTA in a solution is to be calculated in shampoo, a back titration procedure is followed where an excess amount of a magnesium solution of known concentration is added to the EDTA solution and the unreacted magnesium is titrated with a standardized EDTA solution following the direct titration procedure.