Chemical Quality Control

Lab 1
# Assay of Zinc sulfate content in Octozinc® capsules

Principle:

According to United States Pharmacopoeia (USP-30 2007):

Zinc sulphate is determined by **complexometric titration** using:

- **EDTA(ethylene diamine tetra acetic acid disodium salt)** as titrant.
- **EBT(eriochrome black T)** as indicator.
- **Ammonia buffer** as buffer.

N.B: **Ca^{2+} & Mg^{2+} can be determined by the same principle**

- **EDTA** ⟶ **Ethylene diamine tetra acetic acid disodium salt**
- EDTA is also represented as H_2Y^{2-}
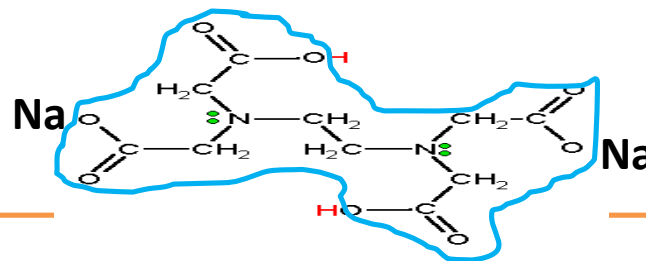
- **EDTA** is prepared as **Molar solution :**
As **It reacts in ratio 1:1 with metals**

- **EBT** is a metallochromic indicator has 2 different colors in complex form with zinc (purple) and in free form (full blue).

Alkaline buffer is used in complexometric titrations .. Why

1. Shift reaction between EDTA and metal forward, to prevent the reversibility of the reaction.

$$H_2Y^{2-} + M^{n+} \rightleftharpoons (MY)^{n-4} + 2H^+$$

$$H_2Y^{2-} + Zn^{2+} \rightleftharpoons (ZnY)^{2-} + 2H^+$$
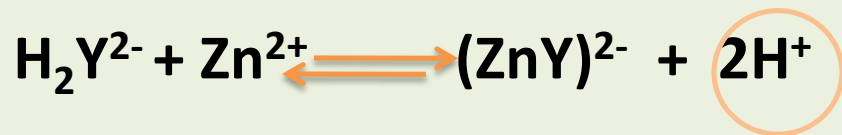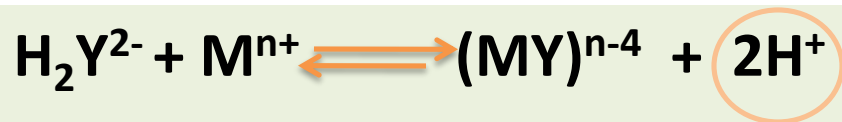
1. Make color change at end point due to change in metal concn. Not due to pH as most metal indicators are also acid base indicators

Precautions during complexometric titration

For sharp end point :
VIGEROUS SHAKING with Rapid titration

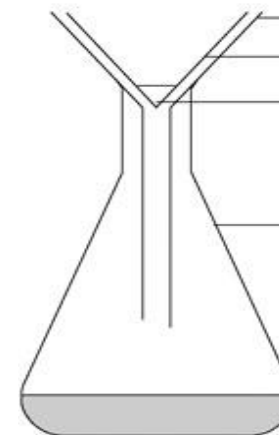✓ **Indicator can be increased any time during titration**

Once end point is reached (free form of indicator), color do NOT change with addition of excess titrant.

Procedure :

1- Transfer the contents of two capsules quantitatively into 100-mL measuring flask

2- Add 50 mL water, shake well for 5 min. for complete extraction, complete to the mark with water, mix and then filter

4- Take 10 mL of the filtrate in conical flask + 2 mL NH_3 buffer + few specks of EBT (purple), and titrate with 0.01 M EDTA till full blue color

3- Discard first 5 mL of the filtrate.

Method Validation is performed to ensure that an analytical method is accurate, specific, reproducible & rugged over a specified range.

For official method

partial validation is required (accuracy, precision and specificity) e.g. Zn SO$_4$.

For non-official method

total validation is required (all validation parameters)

✓ **Accuracy:**

- It the measure of exactness of an analytical method or the closeness of agreement between the measured value and the value that is accepted either as a conventional, true value or an accepted reference value.

- Expressed as **mean Recovery% or Recovery %.**

✓ **Precision:**

- It is the measure of the degree of repeatability of analytical method.

- Expressed as

  relative standard deviation or standard deviation.

Low accuracy
High precision

High accuracy
Low precision

High accuracy
High precision

Calculation:

Each 1mL of 0.01 M EDTA equivalent to 0.002874 gm = *2.874* mg ZnSO$_4$ ⟶ F

Found Amount Of ZnSO$_4$ (in 2 capsules)$= \dfrac{mLs \times f \times F \times 100}{10} =$ mg/2cap

Claimed amount of zinc sulfate in two capsules is <u>**220**</u> mg

Calculate:

ACCURACY:

Expressed as mean recovery %

Recovery % = $\dfrac{found}{claimed} \times 100 = \dfrac{conc.}{220} \times 100 = \quad \%$

Precision:

Expressed as relative standard deviation or standard deviation.

$$\mathbf{RSD} = \dfrac{\mathbf{SD}}{\textbf{Mean recovery}} \mathbf{*100}$$

mls of titration	Amount found	R % (x)	Deviation $\lvert x - x' \rvert$
		100	0
		99	1
		101	1
		95	5
x'			
SD			

rejection rule !!!

If any recovery has its D > 4 D' it must be rejected out of calculation

4D' = 2.66, therefore 5 bigger than 4D'
Therefore rejected

STEPS:

1. Calculate mean recovery (X`) ignoring the suspected one.
2. Calculate |X –X`| for each one.
3. Calculate average deviation (D`) with ignoring deviation of suspected recovery.

If any recovery has **its D > 4 D'** *it must be rejected out of calculation*