Determination of Calcium/Magnesium sample

Uses

- ❑ **Calcium and magnesium supplement**

Principle

Ca^{2+} Only

- At pH =12 murexide form complex with calcium only while magnesium ppt as $Mg(OH)_2$.
- If solution contain both calcium and magnesium is treated with murexide and then titrated with EDTA only calcium will be measured

End point: Pink (metallized form)

↓

Purple (free form)

Total Ca^{2+}/ Mg^{2+}

- If EBT is added to a solution containing calcium and magnesium and the solution is titrated with EDTA (in ammonia buffer),

calcium will react first followed by Mg^{2+} from its complex (Mg-EBT).

- This would be a measure of both calcium and magnesium

End point: Wine red

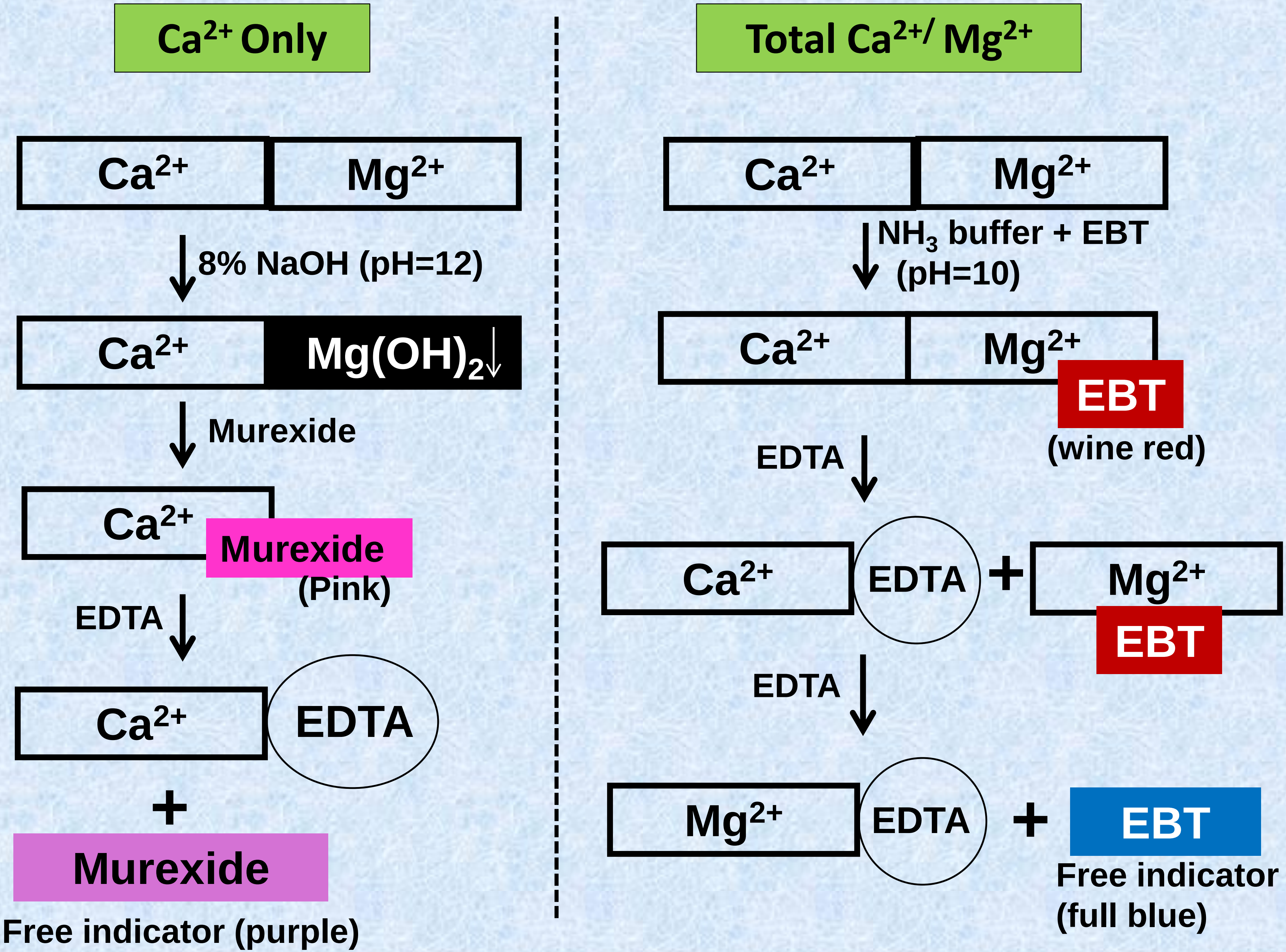
↓

Blue+~~violet tinge~~

↓

Full Blue

- Ca/EDTA complex is more stable than Mg/EDTA complex



Ca2+ Only
Ca2+
Mg2+
8% NaOH (pH=12)
Ca2+
Mg(OH)2↓
Murexide
Ca2+
Murexide
(Pink)
EDTA
Ca2+
EDTA
+
Murexide
Free indicator (purple)
Total Ca2+/ Mg2+
Ca2+
Mg2+
NH3 buffer + EBT
(pH=10)
Ca2+
Mg2+
EBT
(wine red)
EDTA
Ca2+
EDTA
+
Mg2+
EBT
EDTA
Mg2+
EDTA
+
EBT
Free indicator
(full blue)

In Ca^{2+}/ Mg^{2+} sample, Ca^{2+} is determined at pH= 12 .. Why?

1) Mg^{2+} will be precipitated as $Mg(OH)_2$, so removal of interfering ion by separation (precipitation)

2) pH 12 gives maximum stability for Ca/EDTA complex

2- Procedure

Ca^{2+} Only **In Conical Flask**

10 ml Sample
+ 1 ml 8% NaOH
+ few specks Murexide (Pink color)
Titrate against 0.01M EDTA
End point: Purple mls A

Total Ca^{2+}/ Mg^{2+} **In a <u>new</u> Conical Flask**

10 ml Sample
+ 2 ml NH_3 buffer
+ few specks EBT (Wine Red color)
Titrate against 0.01M EDTA
End point: Full blue mls B

3- Calculation

Conc. of $Ca^{2+} = \frac{mlsA \ x \ f \ xF \ x \ 1000}{10} =$ g/L

$F_{Ca^{2+}}$ = 0.00111 g

Conc. of $Mg^{2+} = \frac{(mlsB - mlsA) \ x \ f \ xF \ x \ 1000}{10} =$ g/L

$F_{Mg^{2+}}$ = 0.00246 g

Determination of Aluminium sample

1- Principle

Indirect Complexometry

Al^{3+} form <u>strong</u> aqua complex which needs <u>heating</u> to be broken in order to react with EDTA, so Al^{3+} determined by Back titration

Remaining unreacted EDTA → **Titrated at pH=10 against standard 0.01M $ZnSO_4$**

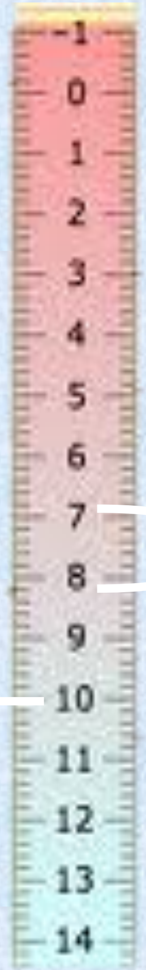
Al^{3+} Sample

Known excess standard EDTA

Amount of EDTA consumed in the quantitative reaction with Al^{3+} (pH needed =7- 8)

Using <u>EBT</u> as indicator
End point: Full blue ⟶ Wine red
Free form — metallized form

pH

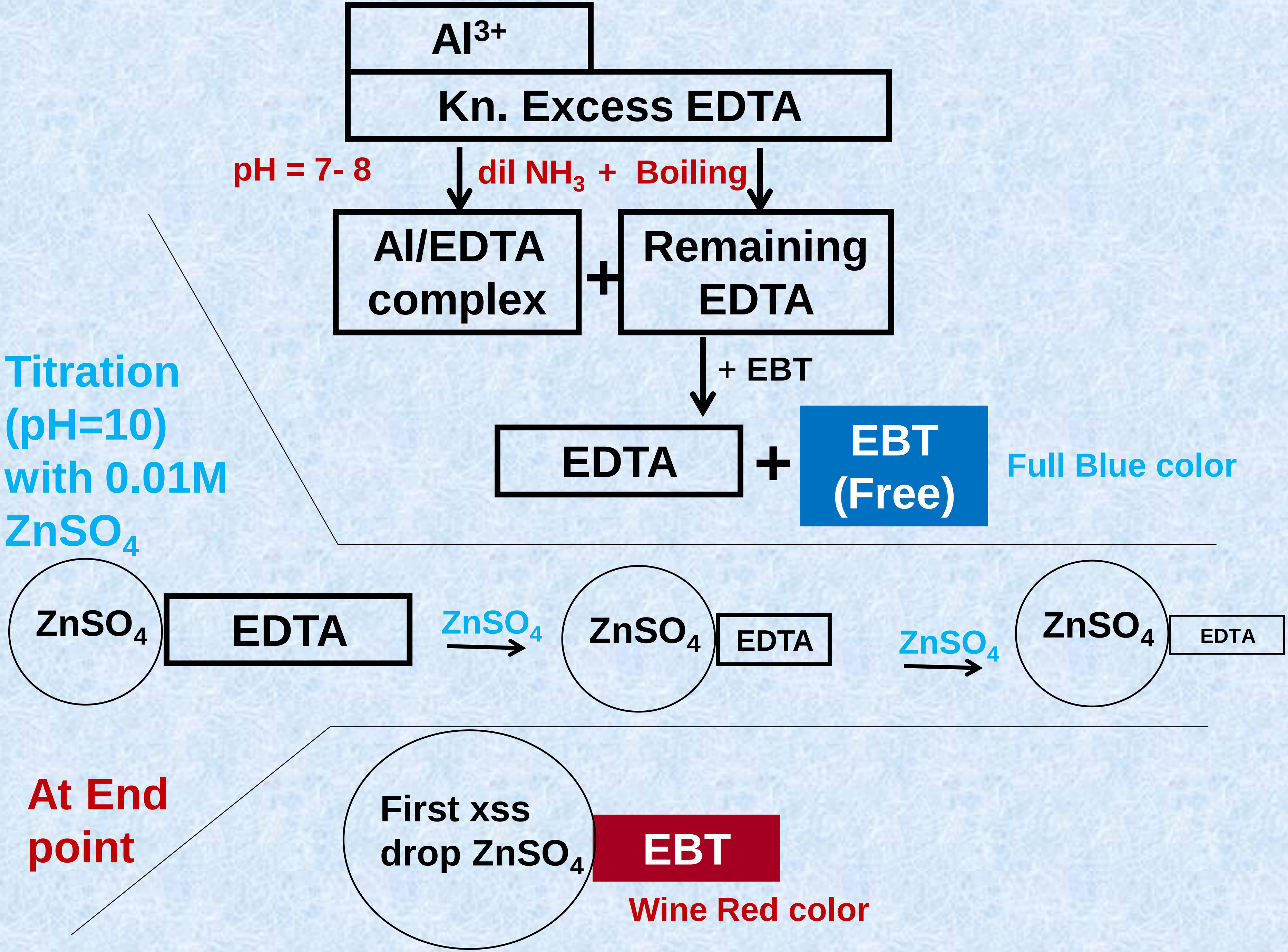


Reaction between EDTA and $ZnSO_4$ is done at pH= 10 (using NH_3 buffer)

To give the maximum stability for Zn/EDTA

Reaction between Al^{3+} and EDTA is done at pH= 7- 8 (using dil NH_3)

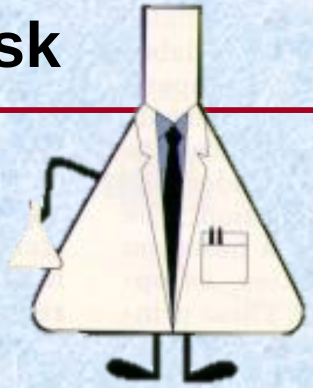
1) To give the maximum stability for Al/EDTA
2) If pH 10 is used, Al^{3+} will be precipitated as $Al(OH)_3$



Al3+
Kn. Excess EDTA
pH = 7- 8
dil NH3 + Boiling
Al/EDTA complex
+
Remaining EDTA
+ EBT
EDTA
+
EBT (Free)
Full Blue color
Titration (pH=10) with 0.01M ZnSO4
ZnSO4
EDTA
ZnSO4
ZnSO4
EDTA
ZnSO4
ZnSO4
EDTA
At End point
First xss drop ZnSO4
EBT
Wine Red color

2- Procedure

In Conical Flask



10 ml Sample
+ 25 ml 0.01M EDTA
+ 4 dps dil NH_3 ⟶ to adjust pH at 7- 8
+ Boil for 10 min.

- ✓ put a funnel on the conical flask to minimize evaporation
- ✓ Once the flask boils, lower the flame

+ Cool well
+ 2ml NH_3 buffer ⟶ to adjust pH at 10
+ few specks EBT (Full Blue color)

Titrate against 0.01M $ZnSO_4$
End point: Wine Red

3- Calculation

1 ml of 0.01 M EDTA = 0.004743 g

Conc. of Al^{3+} = $\frac{(25xf - mls)\ xF\ x\ 1000}{10}$ = g/L

N.B.
$ZnSO_4$ is a primary standard

Mercurimetric determination of Iodide sample

1- Principle

Mercurimetric determination

Iodide may be determined by titration with $Hg(NO_3)_2$.
First a soluble colorless complex $(HgI_4)^{2-}$ is formed.
At the end point, first excess mercuric ions react with the complex forming → red ppt of HgI_2

$$Hg^{2+} + 4I^- \longrightarrow HgI_4^{2-}$$

$$\text{Excess } Hg^{2+} + Hg\,I_4^{2-} \longrightarrow 2HgI_2\downarrow \text{ red ppt}$$

End point: coloreless → First red turbidity

2- Procedure

In Conical Flask

10 ml KI sample
Titrate against N/40 $Hg(NO_3)_2$
End point: first red Turbidity

3- Calculation

$$\textbf{Conc. of iodide} = \frac{ml \times f \times F \times 1000}{10} = \quad g/L$$

F_{I^-} = 0.0083 g

Thank You