Complexometric Titration
Part 1

Dr. Mai Ramadan

Introduction

Review the following definitions:

Complex

Ligand: types

Chelate: Multidentate ligand

Coordination number

$[CuCl_4]^{-2}$ tetrachlorocuprate(II) ion

$[Cu(en)_2]^{+2}$ bisethylenediaminecopper(II) ion

$[Cu(NH_2CH_2COO)_2]$ Bisglycinocopper(II)

Introduction

Titrations based on complex formation called **complexometric titrations.**

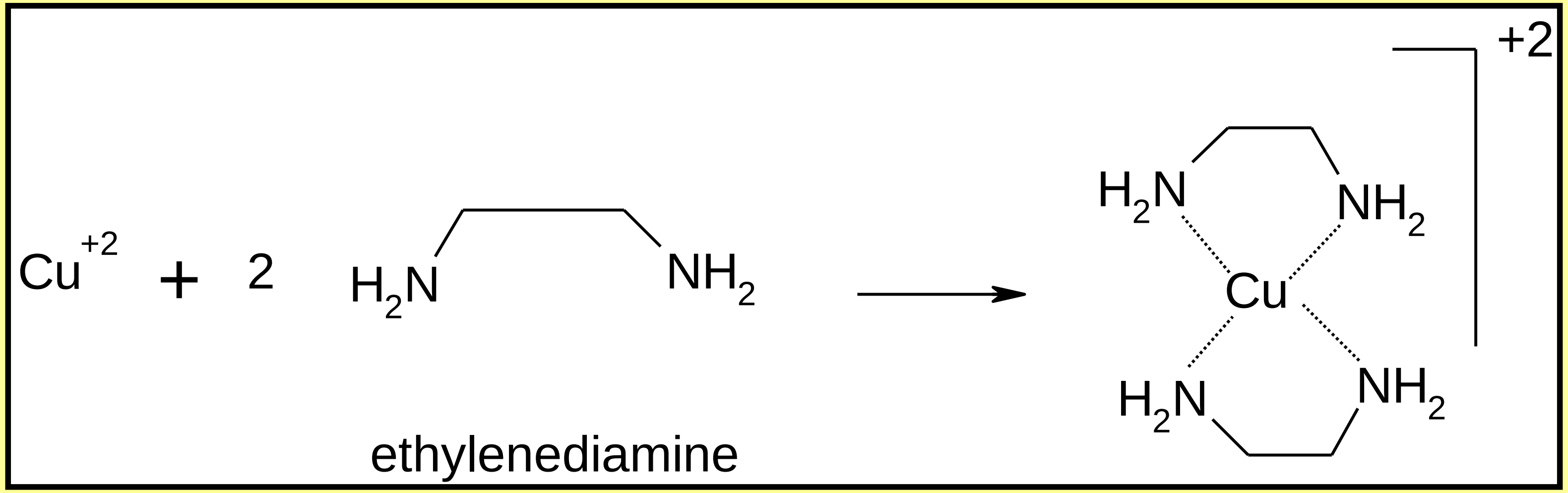
Analytical applications based on a particular class of coordination compounds called **chelates.**

A chelate is produced when a metal ion coordinates with two or more donor groups of a single ligand to form a five- or six-membered heterocyclic ring.

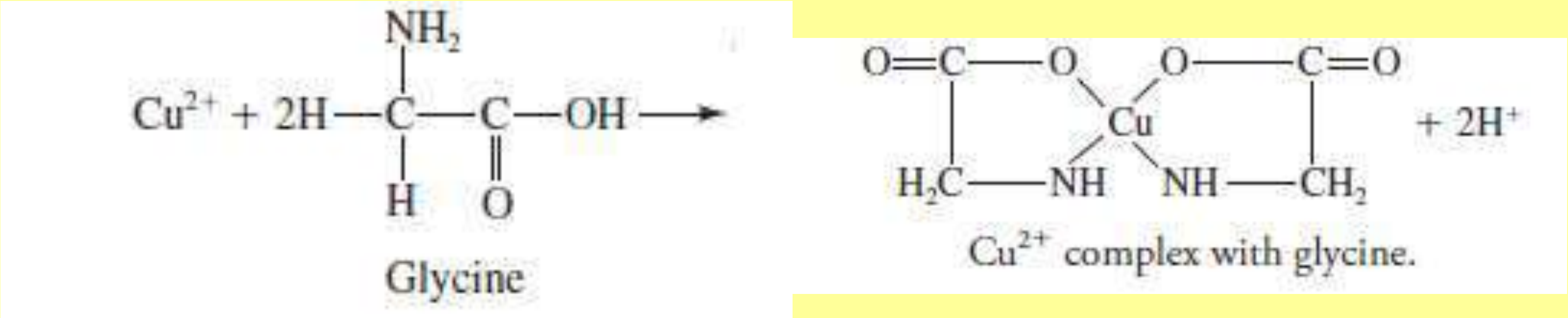
A ligand that has a single donor group, such as ammonia, is called **unidentate** (single-toothed), whereas glycine, which is called **bidentate.**

Tridentate, tetradentate, pentadentate, and hexadentate chelating agents are also known.

Introduction



$[Cu(en)_2]^{+2}$ bisethylenediaminecopper(II) ion



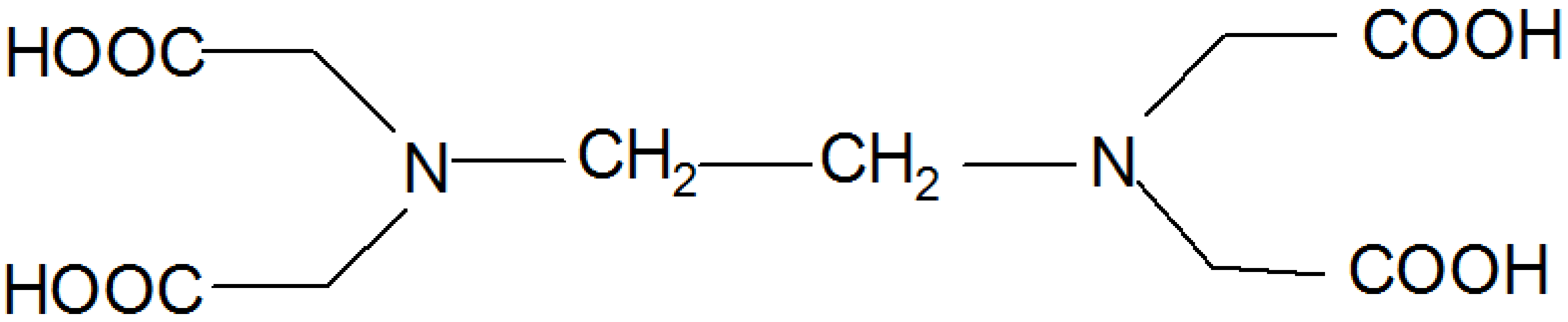
$[Cu\ (NH_2CH_2COO)_2]$ Bisglycinocopper(II)

Organic complexing agents

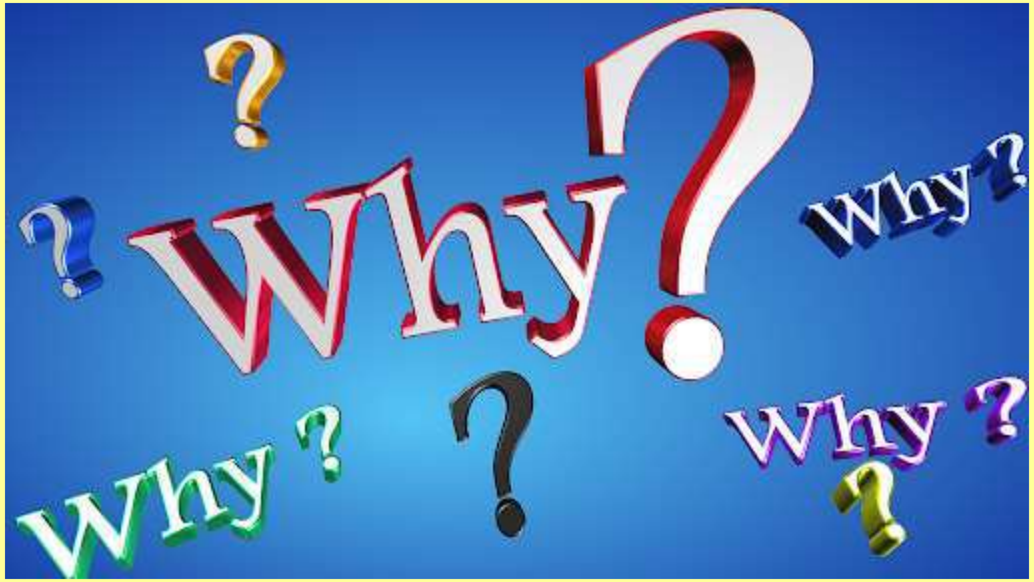
Tertiary amines that also contain carboxylic acid groups form remarkably stable chelates with many metal ions.
Gerold chwarzenbach, a Swiss chemist, first recognized their potential as analytical reagents in 1945. Since his original work, investigators throughout the world have described applications of these compounds to the volumetric determination of most of the metals in the periodic table.

Ethylenediaminetetraacetic Acid (EDTA)

(EDTA)



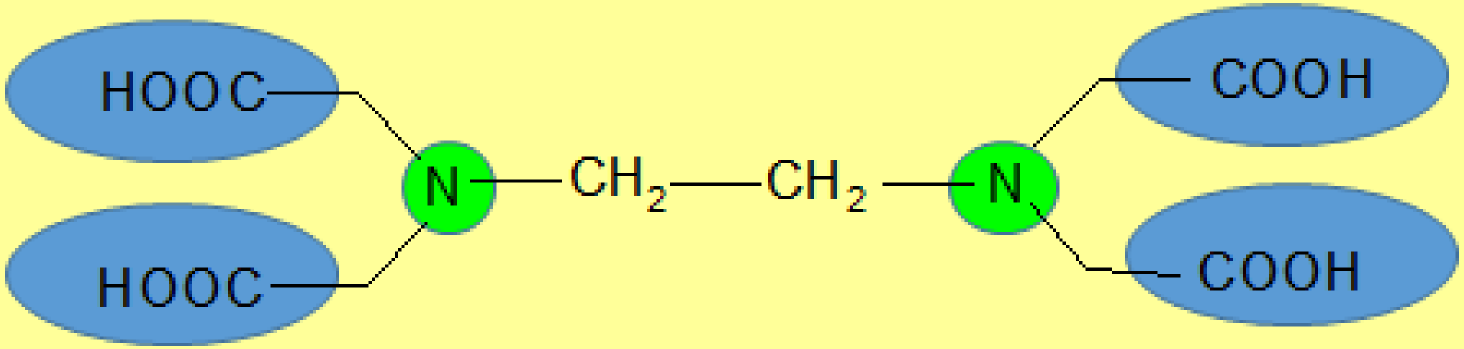
H_4Y



Ethylenediaminetetraacetic Acid (EDTA)????????????

Ethylenediaminetetraacetic Acid (EDTA)

H_4Y



↕ $- H^+$

H_3Y^{-1}

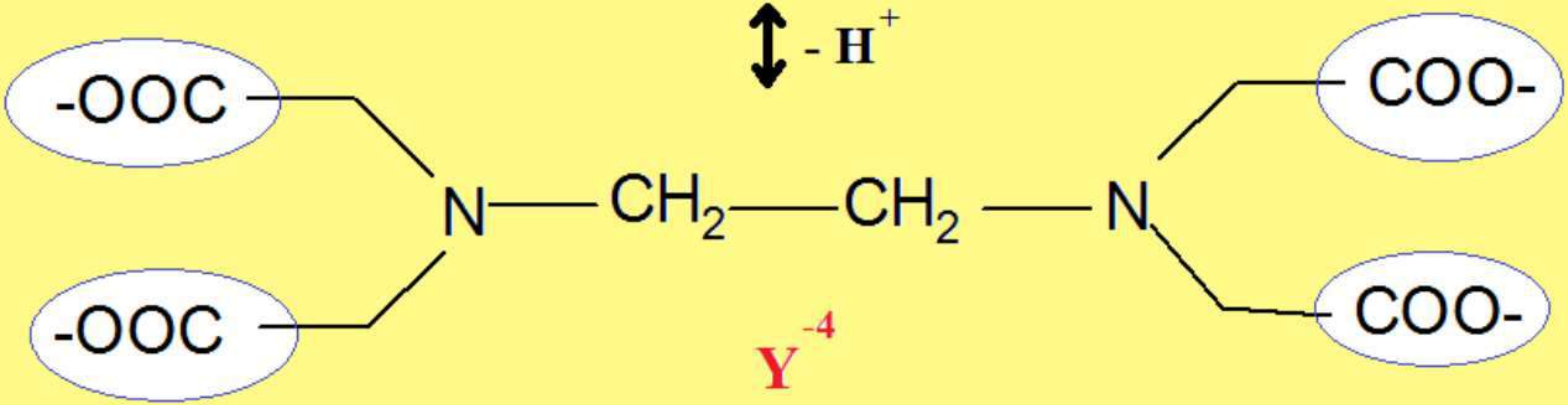
↕ $- H^+$

H_2Y^{-2}

↕ $- H^+$

HY^{-3}

↕ $- H^+$



Y^{-4}

EDTA is Hexadendate ligand

EDTA forms very stable chelate with ions. High K_f

The complexation occurs in one step

M-EDTA complexes are water soluble

The stoichiometry for all metal ions is 1:1 irrespective of the charge

Standard solution preparation

$Na_2H_2Y \cdot 2H_2O$

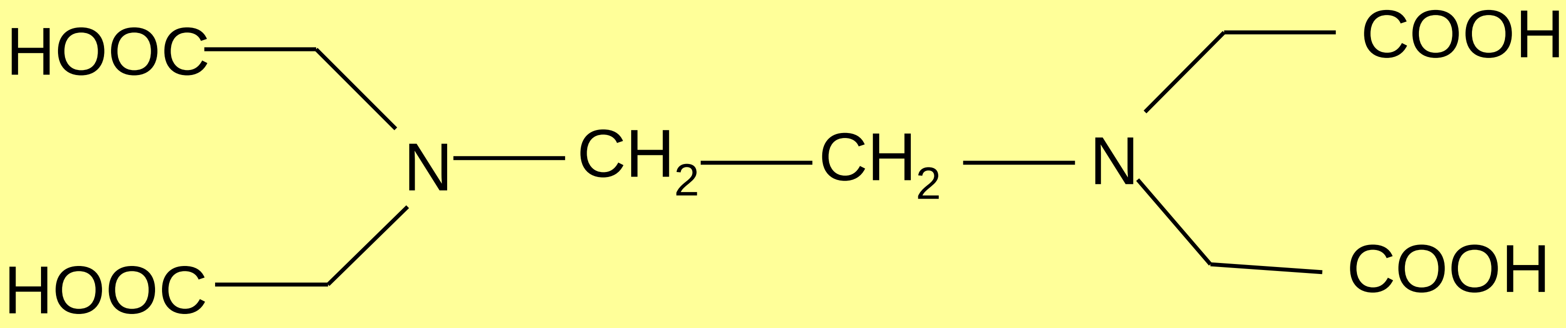
Ethylenediaminetetraacetic Acid (EDTA)

- ☐ EDTA is the most widely used complexometric titrant.
- ☐ EDTA is a hexadentate ligand.
- ☐ It should be simplify as H_4Y.
- ☐ Solution of EDTA are particularly valuable as **titrant** because the EDTA combines with metal ions in a 1:1 ratio regardless of the charge on the cation (+1, +2, +3, +4).

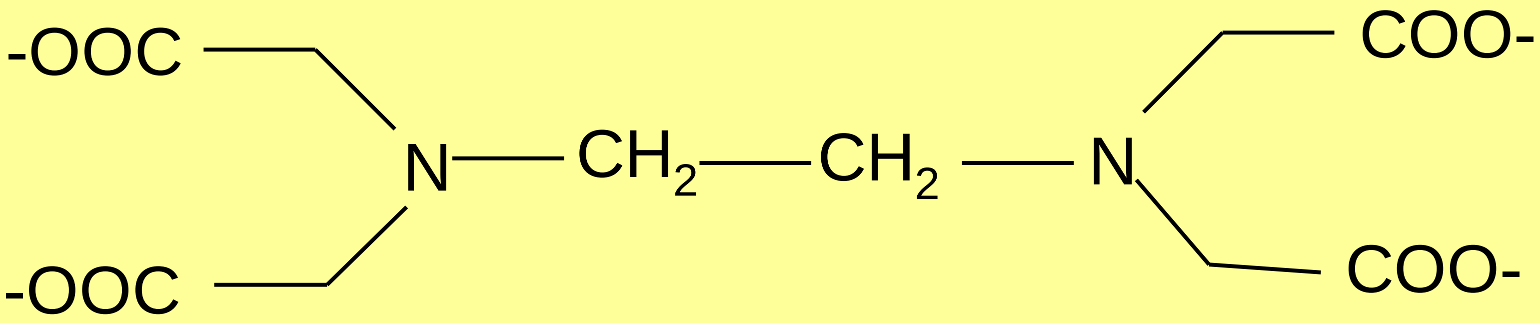
Ethylenediaminetetraacetic Acid (EDTA)

- Standard EDTA solutions: Na_2H_2Y . 2H2O.
- EDTA has acidic character. It loses 4 protons stepwise with dissociation constants **K_1=1.02*10^{-2}, K_2 = 2.14*10^{-3}, K_3 = 6.92*10^{-7}, K_4 = 5.5*10^{-11}.**
- Y^{-4} is predominant at pH higher than 10.

(EDTA)



H_4Y

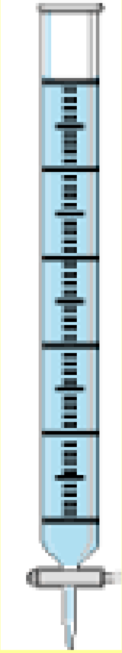


Y^{-4}

PH> 10

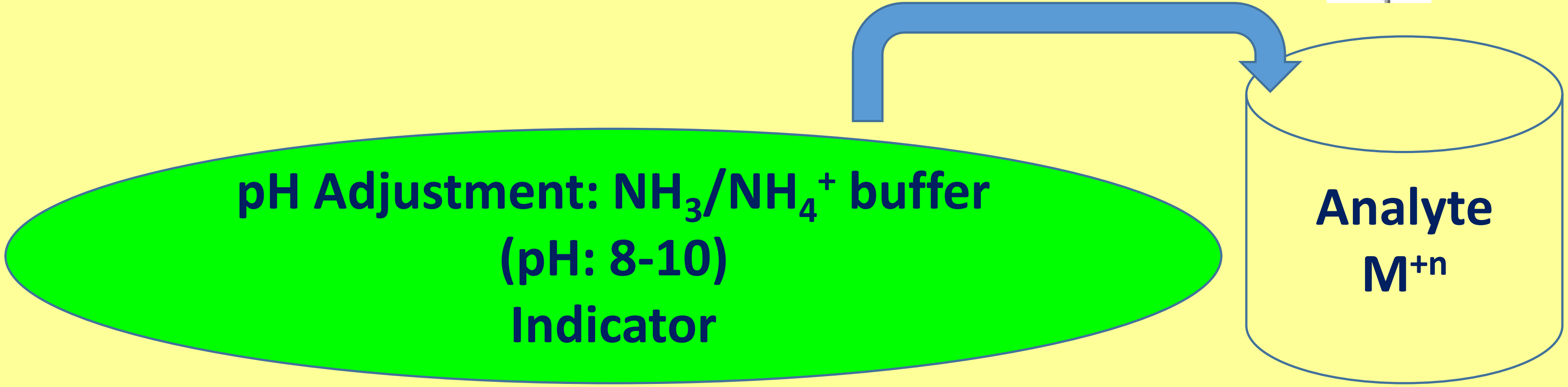
EDTA-titration

$$M^{+n} + H_2Y^{-2} \longrightarrow [MY]^{n-4} + 2\,H^+$$



Titrant

H_2Y^{-2}



pH Adjustment: NH_3/NH_4^+ buffer

(pH: 8-10)

Indicator

Analyte

M^{+n}

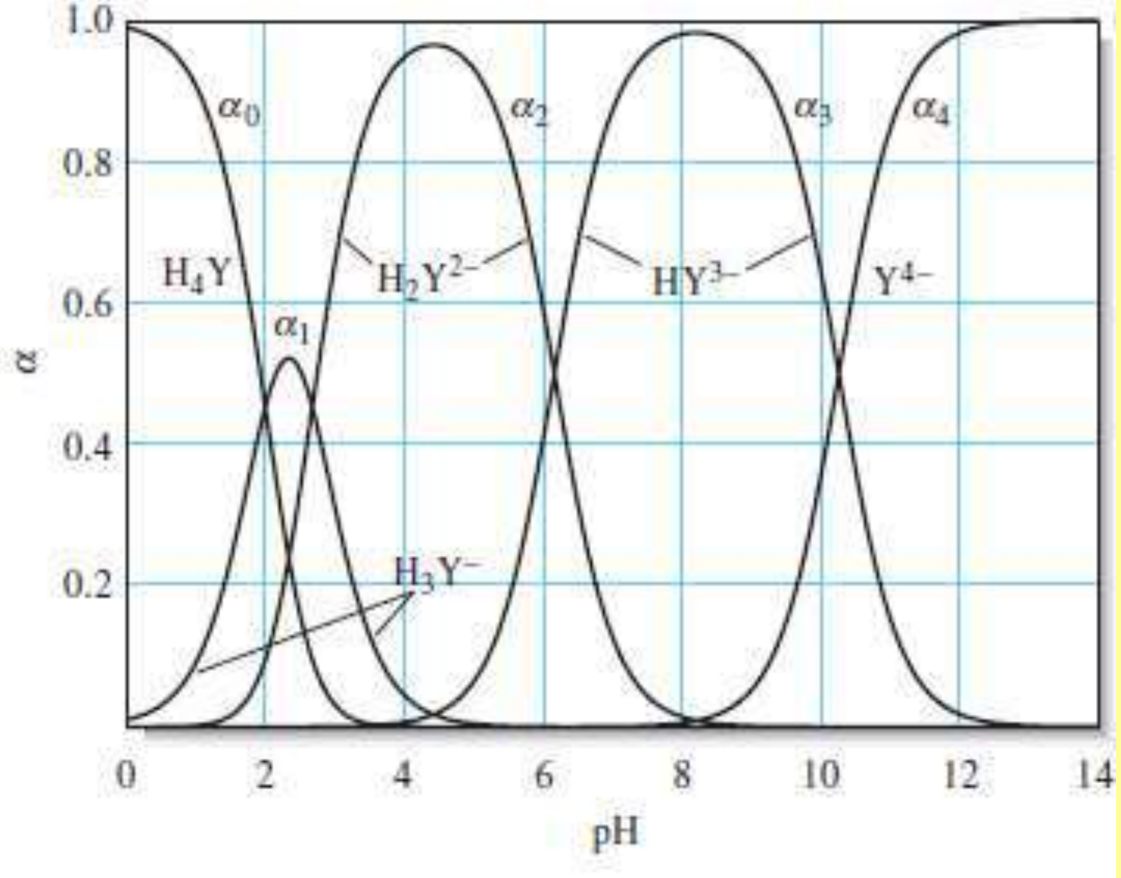
Ethylenediaminetetraacetic Acid (EDTA)

Composition of EDTA solutions as a function of pH.

H4Y is only a major component in very acidic solutions (pH < 3).

pH range 3-10 H2Y^{-2} and HY $^{-3}$ are predominant.

Y^{-4} is a significant component only in basic solutions (pH > 10)



$$c_T = [Y^{4-}] + [HY^{3-}] + [H_2Y^{2-}] + [H_3Y^{3-}] + [H_4Y]$$

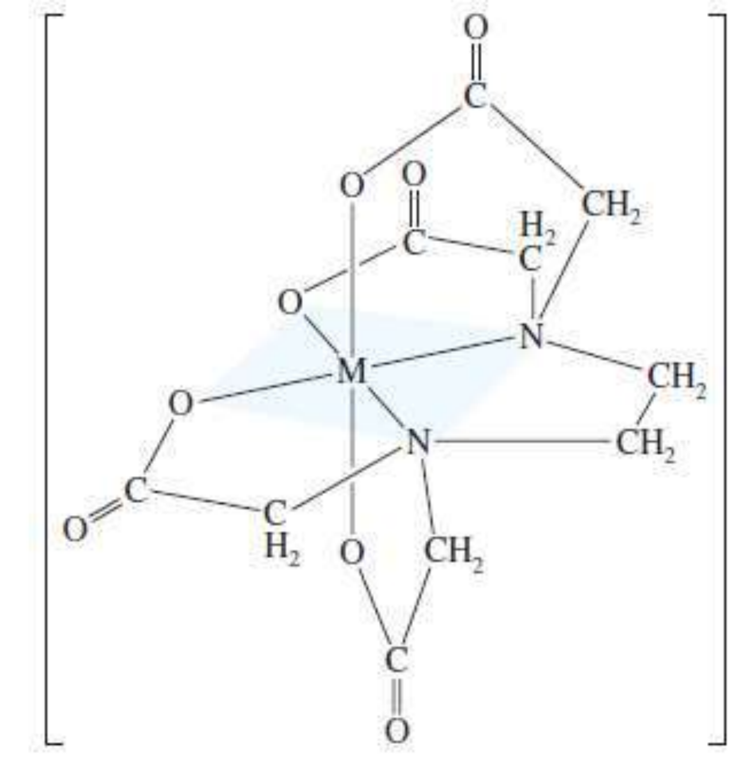
$$\alpha_4 = \frac{[Y^{4-}]}{c_T}$$

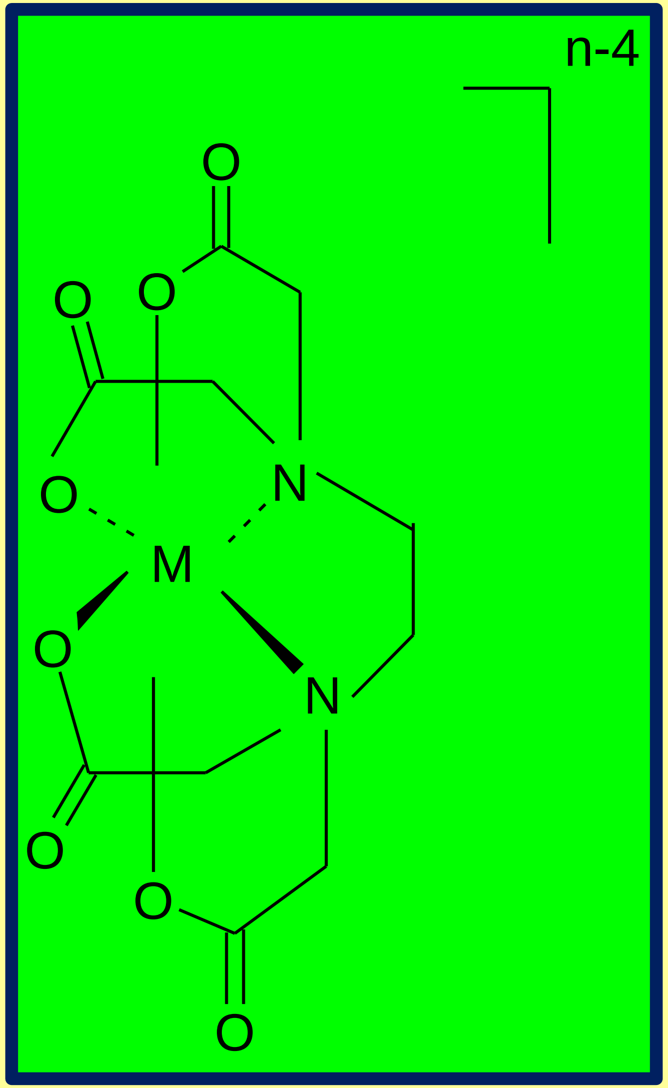
Complexes of EDTA and Metal Ions

$$M^{+n} + Y^{-4} \longleftrightarrow [MY]^{n-4}$$

The complexes of EDTA are soluble and are mainly colorless.

It has **cis-octahedral** geometry

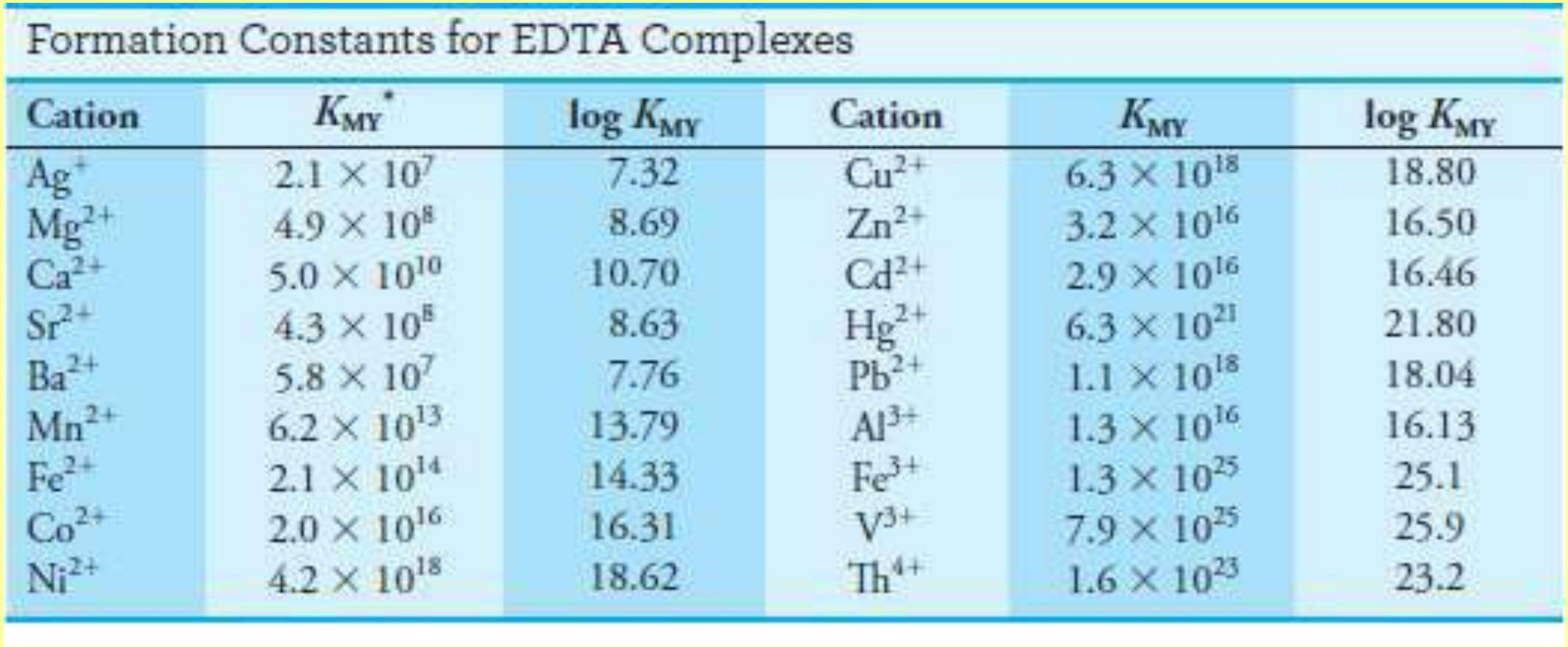




Stability of EDTA complexes

$$M^{n+} + Y^{4-} \rightleftharpoons MY^{(n-4)+} \qquad K_{MY} = \frac{[MY^{(n-4)+}]}{[M^{n+}][Y^{4-}]}$$

The formation constant: K_f



Formation Constants for EDTA Complexes

Cation	K_{MY}*	$\log K_{MY}$	Cation	K_{MY}	$\log K_{MY}$
Ag^+	2.1×10^7	7.32	Cu^{2+}	6.3×10^{18}	18.80
Mg^{2+}	4.9×10^8	8.69	Zn^{2+}	3.2×10^{16}	16.50
Ca^{2+}	5.0×10^{10}	10.70	Cd^{2+}	2.9×10^{16}	16.46
Sr^{2+}	4.3×10^8	8.63	Hg^{2+}	6.3×10^{21}	21.80
Ba^{2+}	5.8×10^7	7.76	Pb^{2+}	1.1×10^{18}	18.04
Mn^{2+}	6.2×10^{13}	13.79	Al^{3+}	1.3×10^{16}	16.13
Fe^{2+}	2.1×10^{14}	14.33	Fe^{3+}	1.3×10^{25}	25.1
Co^{2+}	2.0×10^{16}	16.31	V^{3+}	7.9×10^{25}	25.9
Ni^{2+}	4.2×10^{18}	18.62	Th^{4+}	1.6×10^{23}	23.2

Conditional formation constant

Conditional formation constants are pH dependent K'_{MY}

$$M^{n+} + Y^{4-} \rightleftharpoons MY^{(n-4)+} \qquad K_{MY} = \frac{[MY^{(n-4)+}]}{[M^{n+}]\alpha_4 c_T}$$

$$K'_{MY} = \alpha_4 K_{MY} = \frac{[MY^{(n-4)+}]}{[M^{n+}]c_T}$$

Metals-ion indicator

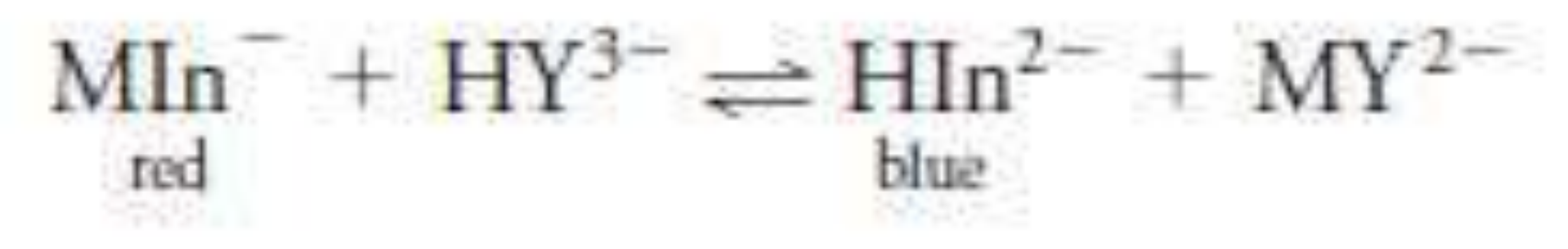
A typical indicator used in titration of many common metals is Erichrom-black T. This indicator contains sulfonic acid group which dissociated completely in water. The other phenolic groups are dissociated partially.

$$H_2In^- \text{ (red)} + H_2O \longleftrightarrow HIn^{-2} \text{ (blue)} + H_3O^+$$

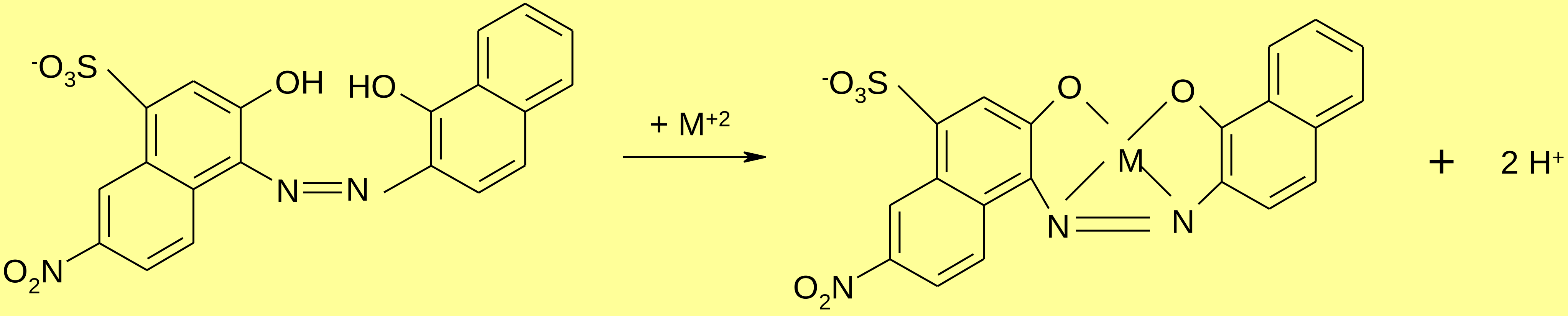
$$HIn^{-2} \text{ (blue)} + H_2O \longleftrightarrow In^{-3} \text{ (orange)} + H_3O^+$$

The metal ion complex of Erichrom-black T is wein red. When the indicator during the titration with EDTA becomes free (not bonded) then colour changes to blue indicating the end point.

Metals-ion indicator



$$\underset{\text{red}}{MIn^{-}} + HY^{3-} \rightleftharpoons \underset{\text{blue}}{HIn^{2-}} + MY^{2-}$$



Erichrom-Black T
H_2In^{-}

Indicator complex of metal ion
MIn^{-}

Metals-ion indicator

Blocking of indicator:

Some cations like Cu^{+2}, Ni^{+2}, Co^{+2}, Cr^{+3}, Fe^{+3} and Al^{+3} are bonded very strongly to erichrom black T and don not dissociate from the indicator complex to react with the titrant EDTA-solution. These cations are saied to ***block*** the indicator. It inhibits a direct titration with EDTA.

Metals-ion indicator

Disadvantages of Erichrom black T:

- Unstable solution on standing
- Used in basic pH
- Complex of cation with indicator in comparison to complex with edta is very unstable that the release of cation can be before equivalence point like Ca+2.
- Other metal ion indicators are murexid, calmagite, xylenol orange.

Complexometric Titration
Part 2

Dr. Mai Ramadan

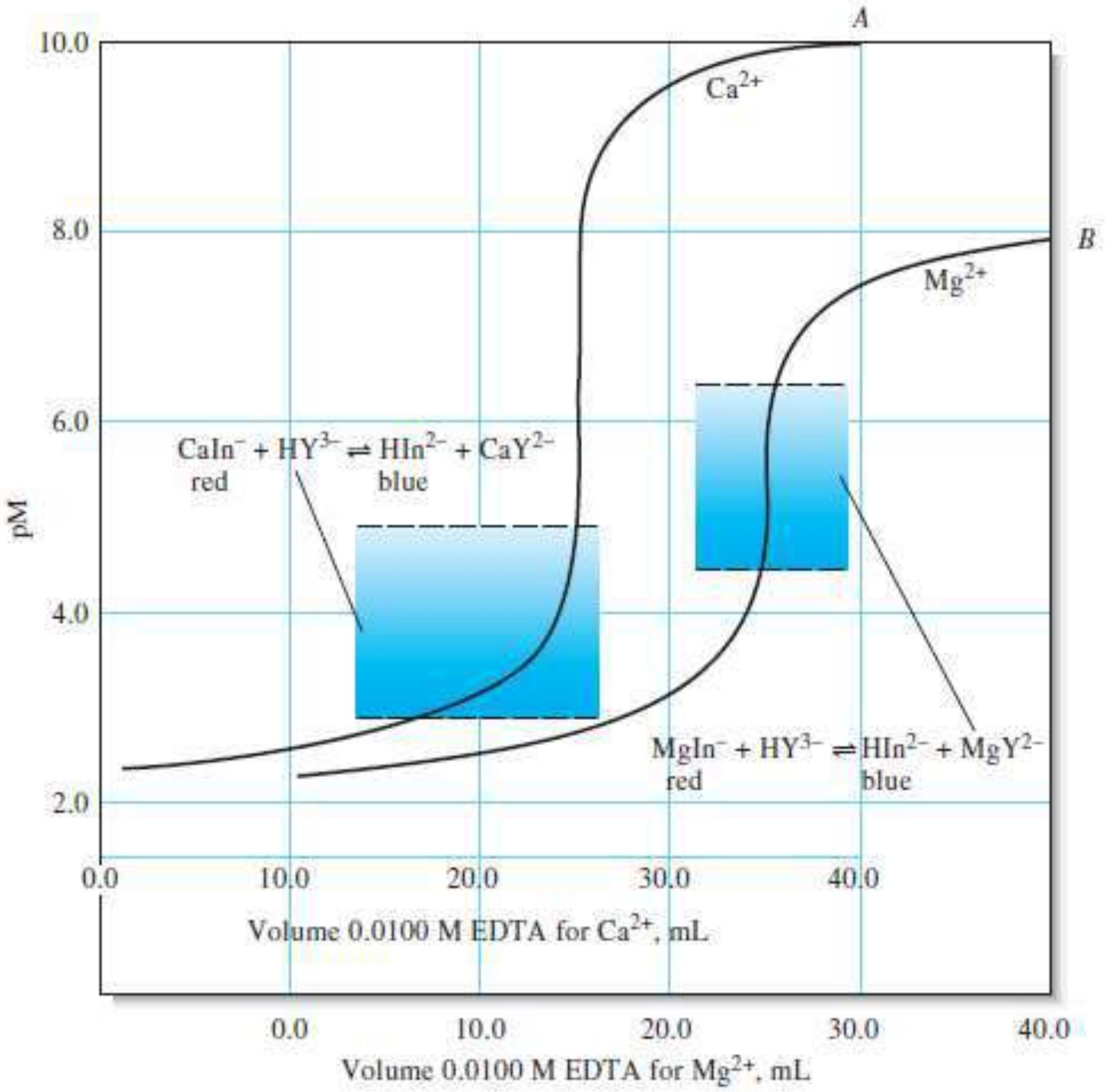
EDTA Titration Curves

EDTA titration curves

for 50.0 mL of 0.005 M

Ca+2 ($K'_{CaY} = 1.75*10^{10}$)

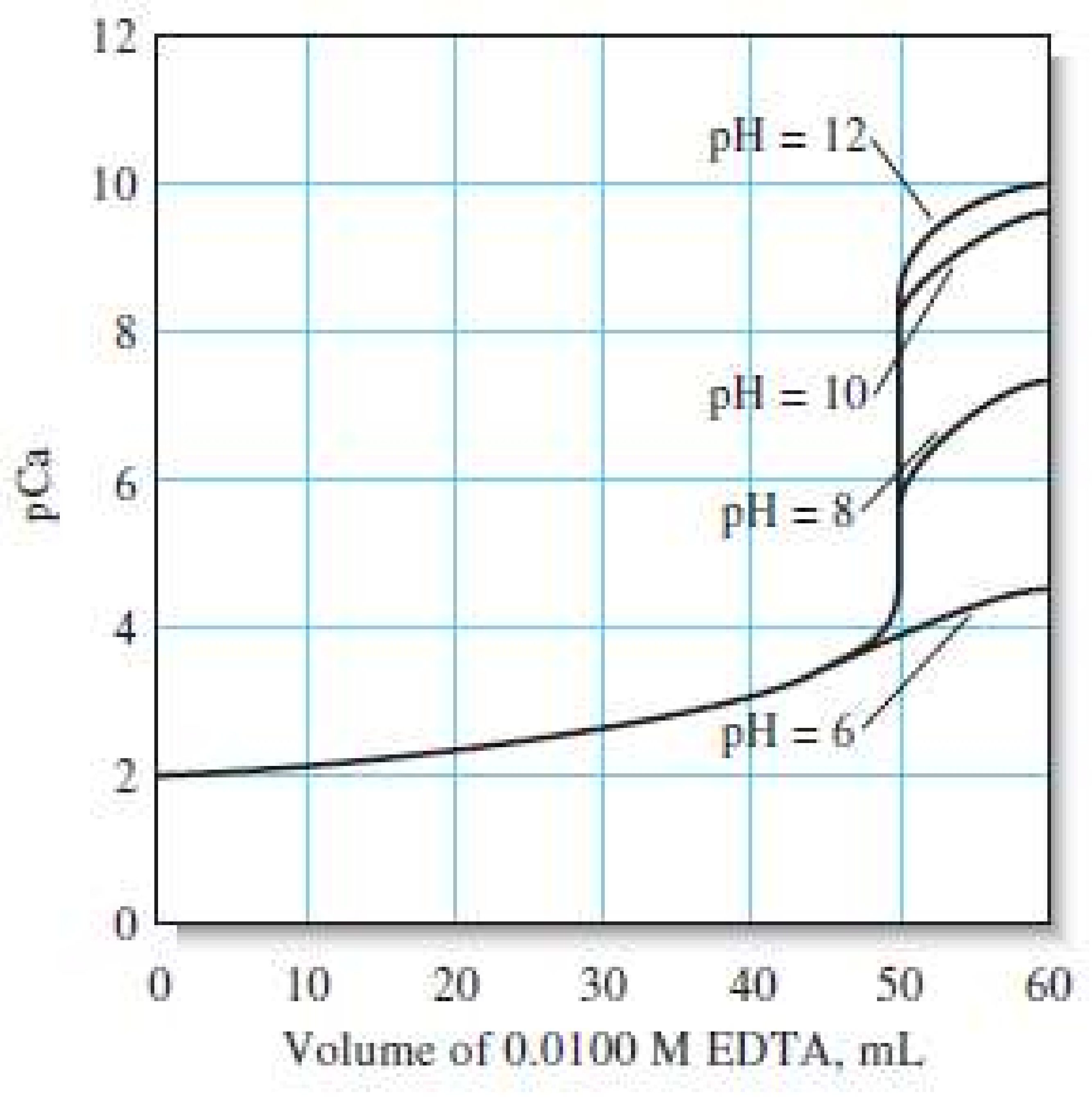
Mg+2($K'_{MgY} = 1.72*10^{8}$) *at pH 10.0.*



EDTA Titration Curves

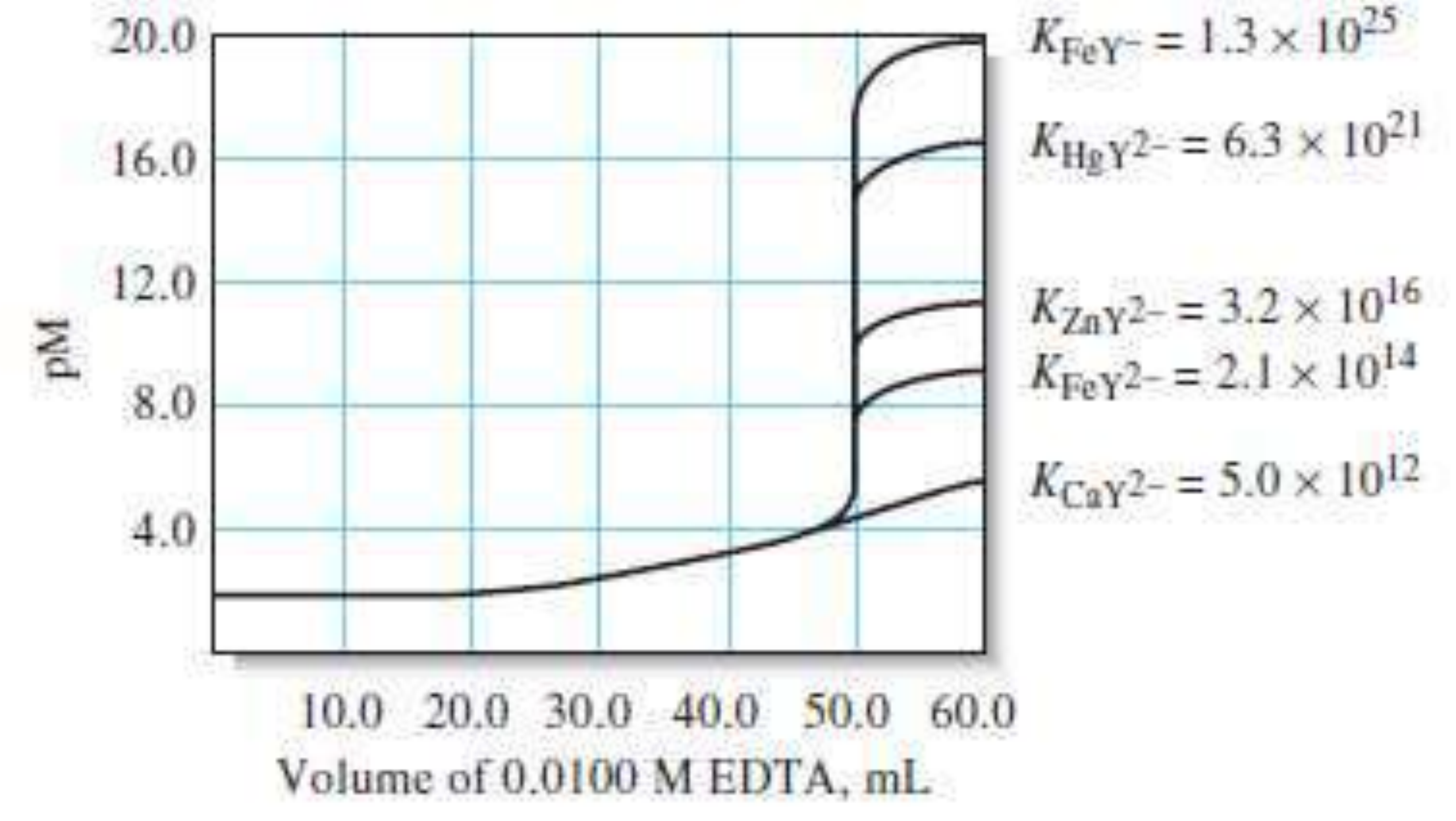
Influence of pH on the titration of 0.01M Ca+2 with 0.01 M EDTA.

Note that the end point becomes less sharp as the pH decreases because the complex-formation reaction is less complete under these circumstances.



EDTA Titration Curves

Titration curves for 50.0 mL of 0.01 M solutions of various cations at pH 6.0.



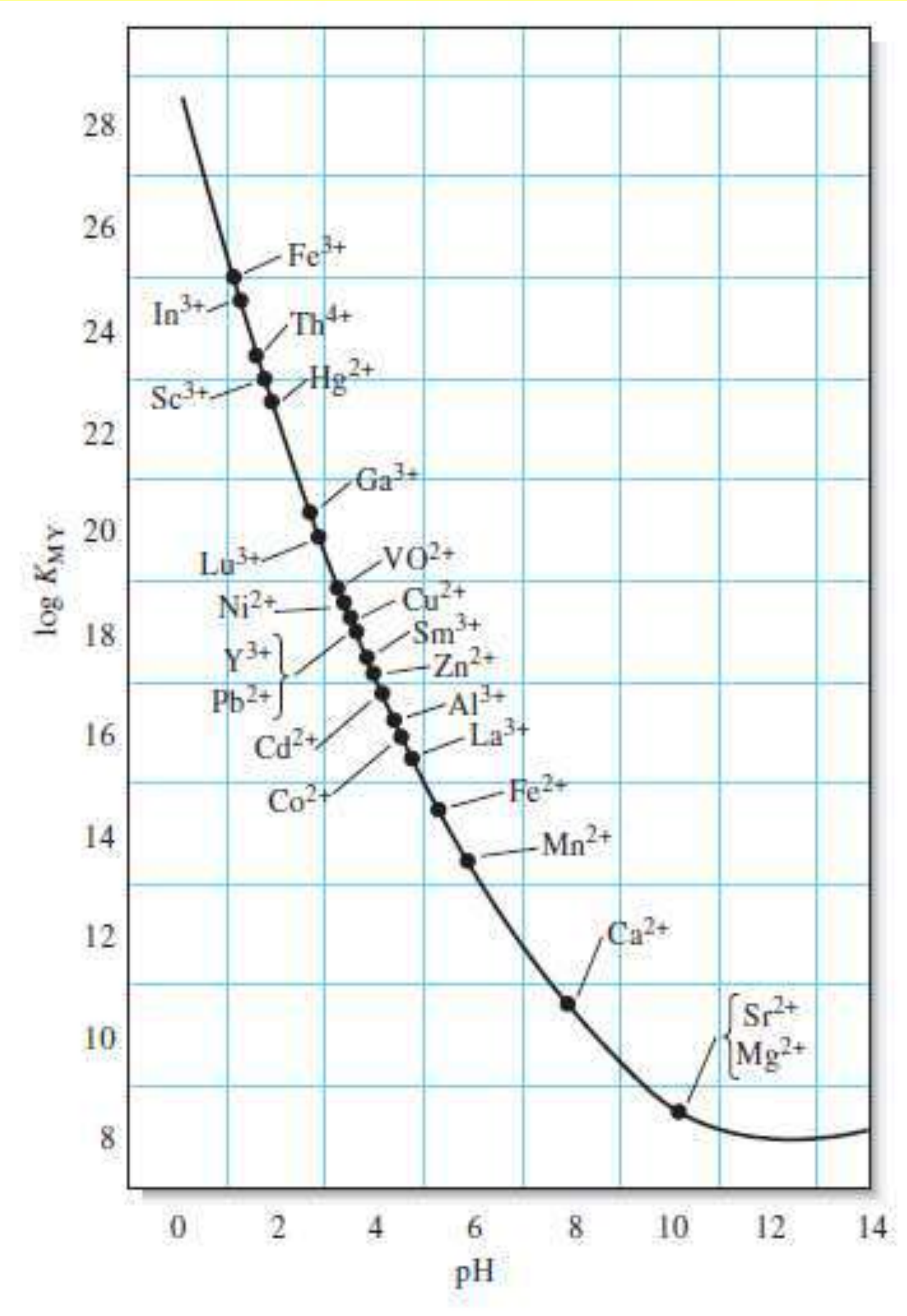
EDTA Titration Curves

Minimum pH needed

for satisfactory titration

of various cations

with EDTA.



Auxiliary complexing agents

Many cations form hydrous oxide precipitates (hydroxides, oxides, or oxyhydroxides) when the pH is raised to the level required for their successful titration with EDTA.

Auxiliary complexing agents must be used in EDTA titrations to prevent precipitation of the analyte as a hydrous oxide.

For example, Ammonia buffer is added to zinc(II) for EDTA titration. The buffer adjust a pH that ensures complete reaction between cation and titrant. **In addition**, ammonia forms ammine complexes $[Zn(NH_3)]^{+2}$, $[Zn(NH_3)_2]^{+2}$, $[Zn(NH_3)_3]^{+2}$ and prevents formation of $Zn(OH)_2$ which has a $K_{sp} = 3.0*10^{-16}$, particularly in the early stages of the titration.

Titration Methods Involving EDTA

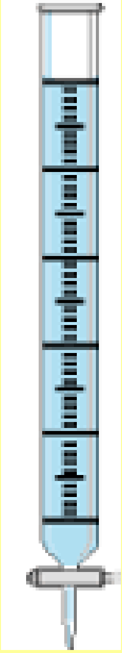
Direct titration

The solution of an analyte (cations) is buffered to a suitable pH then the indicator is added following which the titrant EDTA-standard solution is allowed to react with the analyte. To prevent the precipitation of some cations auxiliary complexing agent is added like NH_3 for Zn^{+2} and tartrate for Pb^{+2}.

Direct titration

$$M^{+n} \quad + \quad H_2Y^{-2} \longrightarrow [MY]^{n-4} \quad + \quad 2\,H^+$$

End Point: Wein red color ⟶ blue



Titrant
H_2Y^{-2}

pH Adjustment: NH_3/NH_4^+ buffer
(pH: 8-10)
Erichrom black T

Analyte
M^{+n}

Titration Methods Involving EDTA

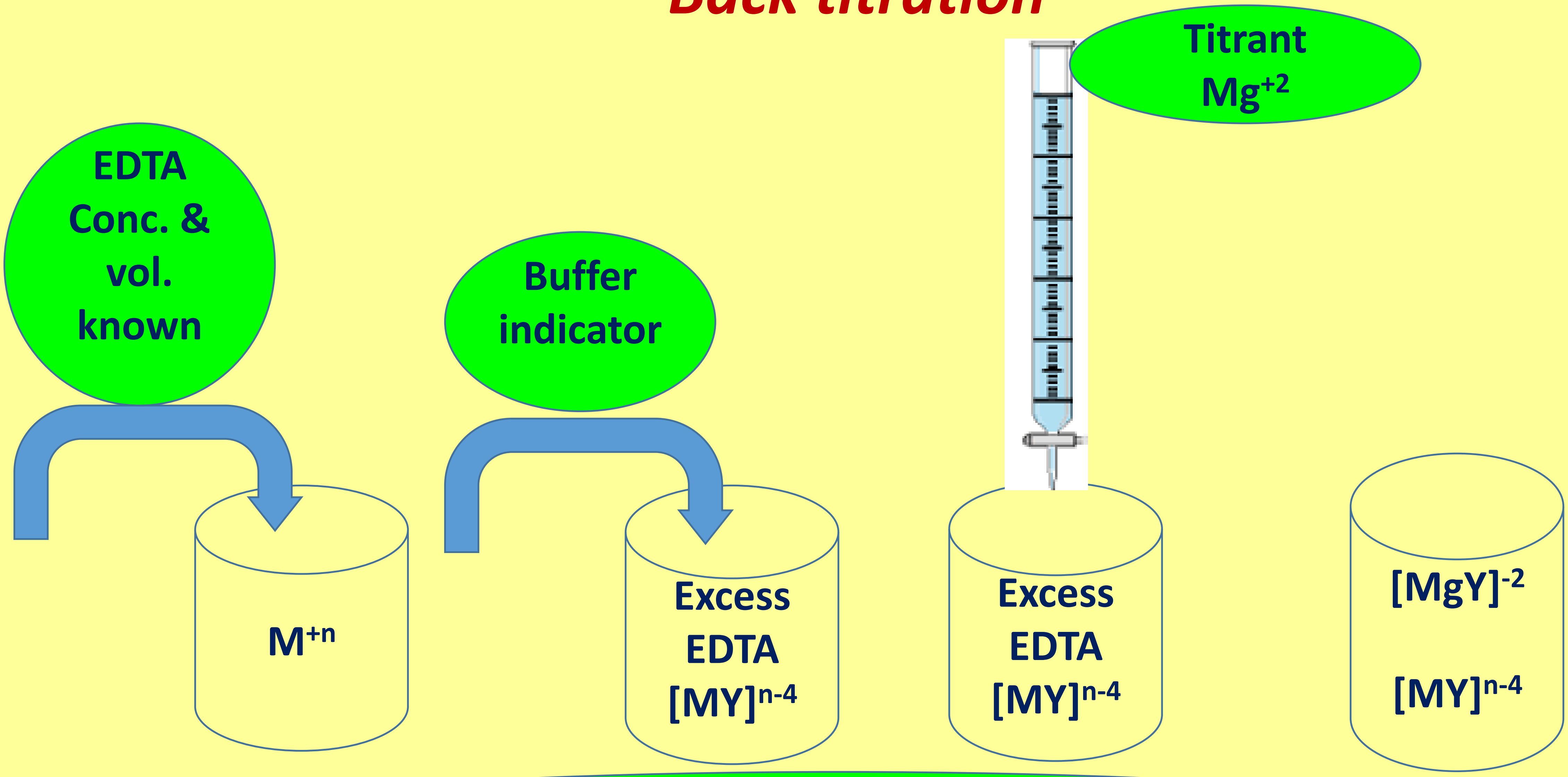
Back titration

A solution of the analyte (cation) is treated with an excess of EDTA standard solution, following which the excess EDTA is determined by a titration using Mg^{+2} solution.

Back titration is required when the reaction of analyte such as Cr(III) and Co(III) with EDTA is too slow, the analyte blocks the indicator or the analyte precipitated.

For this procedure to be successful, it is necessary that the magnesium or zinc ions form an EDTA complex that is less stable than the corresponding analyte complex.

Back titration



EDTA Conc. & vol. known

Buffer indicator

Titrant Mg^{+2}

M^{+n}

Excess EDTA $[MY]^{n-4}$

Excess EDTA $[MY]^{n-4}$

$[MgY]^{-2}$

$[MY]^{n-4}$

End point when Erichrom black T is the indicator:
Blue (Free indicator) -------------- Wein red (complex of $[MgIn]^{-1}$

Back titration

$$M^{+n} + H_2Y^{-2}_{reacted} \longrightarrow [MY]^{n-4} + 2\,H^{+}$$

$$Mg^{+2}_{titrant} + H_2Y^{-2}_{excess} \longrightarrow [MgY]^{-2} + 2\,H^{+}$$

Why was the titrant Mg^{+2}?

Is there is a possibility that Mg^{+2} reacts with EDTA in $[MY]^{n-4}$????

Hint

Remember the K_f values

Titration Methods Involving EDTA

Displacement titration

The analyte solution is treated with an excess of $[MgY]^{-2}$. The analyte displaces Mg^{+2} from the complex, which is then titrated with EDTA solution.

$$M^{+n} + [MgY]^{-2} \longrightarrow [MY]^{n-4} + Mg^{-+2}$$

M^{+n} is like Hg^{+2}. The K_f of $[HgY]^{-2} >> [MgY]^{-2}$

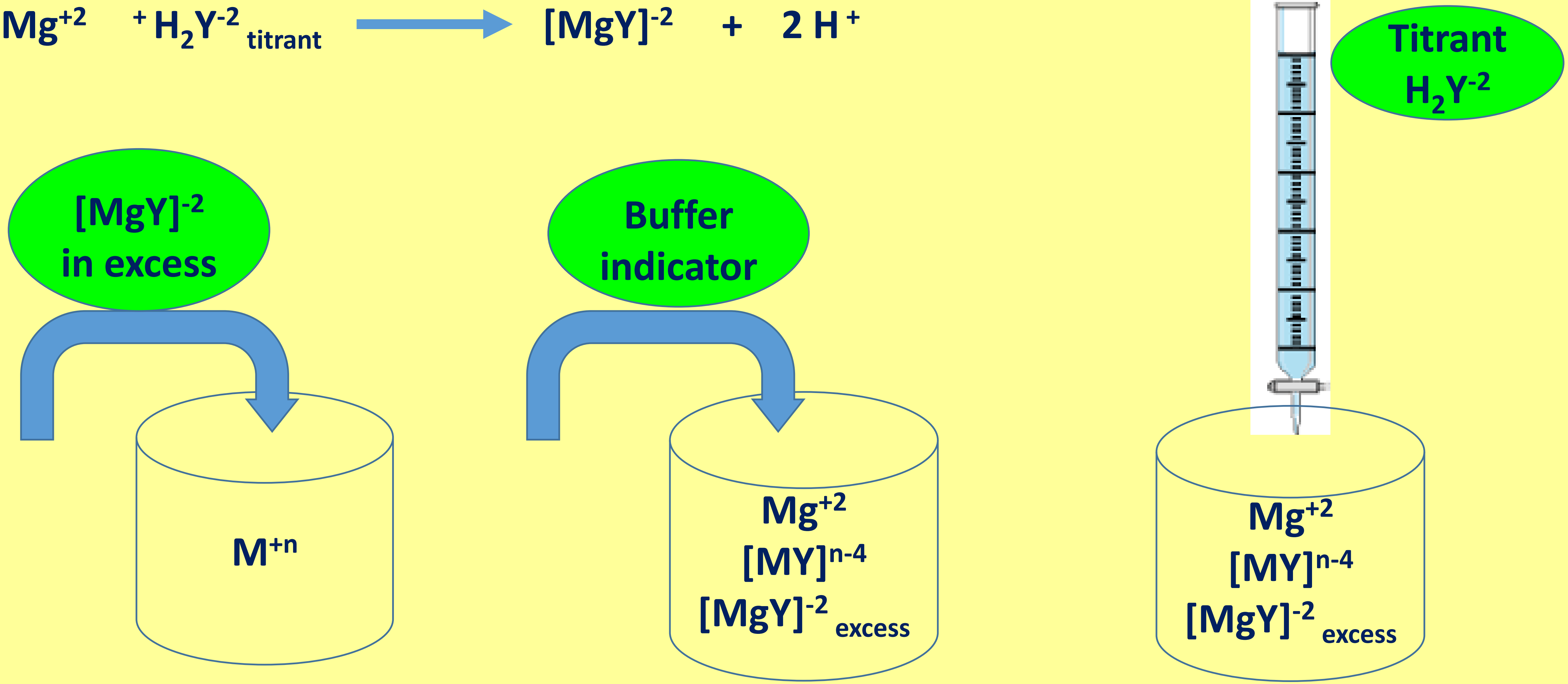
Determination of Ag^{+}: To Ag+ solution tetracyanonickelate(II) complex is added. The liberated Ni^{+2} is then titrated with EDTA.

$$2\,Ag^{+} + [Ni(CN)_4]^{-2} \longrightarrow 2\,[Ag(CN)_2]^{-} + Ni^{+2}$$

Displacement titration

$$M^{+n} + [MgY]^{-2} \longrightarrow [MY]^{n-4} + Mg^{-+2}$$

$$Mg^{+2} + H_2Y^{-2}{}_{\text{titrant}} \longrightarrow [MgY]^{-2} + 2H^{+}$$



$[MgY]^{-2}$ in excess

M^{+n}

Buffer indicator

Mg^{+2}

$[MY]^{n-4}$

$[MgY]^{-2}{}_{\text{excess}}$

Titrant H_2Y^{-2}

Mg^{+2}

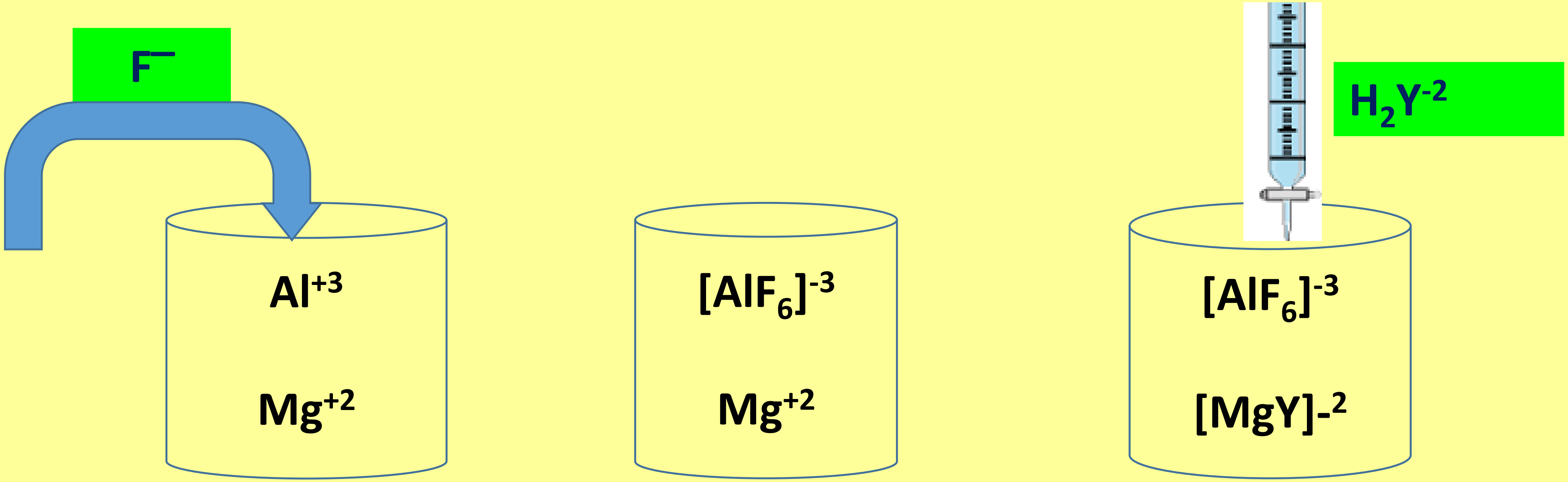
$[MY]^{n-4}$

$[MgY]^{-2}{}_{\text{excess}}$

Titration Methods Involving EDTA

Masking and demasking

A masking agent is a complexing agent that reacts selectively with a component in a solution, so it is prevented to interfere with other components.



F^-

Al^{+3}

Mg^{+2}

$[AlF_6]^{-3}$

Mg^{+2}

H_2Y^{-2}

$[AlF_6]^{-3}$

$[MgY]^{-2}$

Flouride ion is a masking agent for Al^{+3}, which will not interfere with EDTA titration.

$$Al^{+3} + 6\,F^- \longrightarrow [AlF_6]^{-3}$$

Titration Methods Involving EDTA

Masking and demasking

- ❑ CN- is a masking agent for many cations like Co^{+2}, Ni^{+2}, Zn^{+2}, Pd^{+2}, Ag^{+}, Fe^{+2}, Cd^{+2} but it does not with others like Mg^{+2}, Ca^{+2}, Mn^{+2} and Pb^{+2}.
- ❑ BAL (2,3-dimercapto-1-propanol) for Pb^{+2}
- ❑ Hexamethylene-tetramine for Cr^{+3}.

Titration Methods Involving EDTA

Masking and demasking

$$Cd^{+2} \quad + 4\ CN^{-} \longrightarrow [Cd(CN)_4]^{-2}$$

masking agent **stable complex**

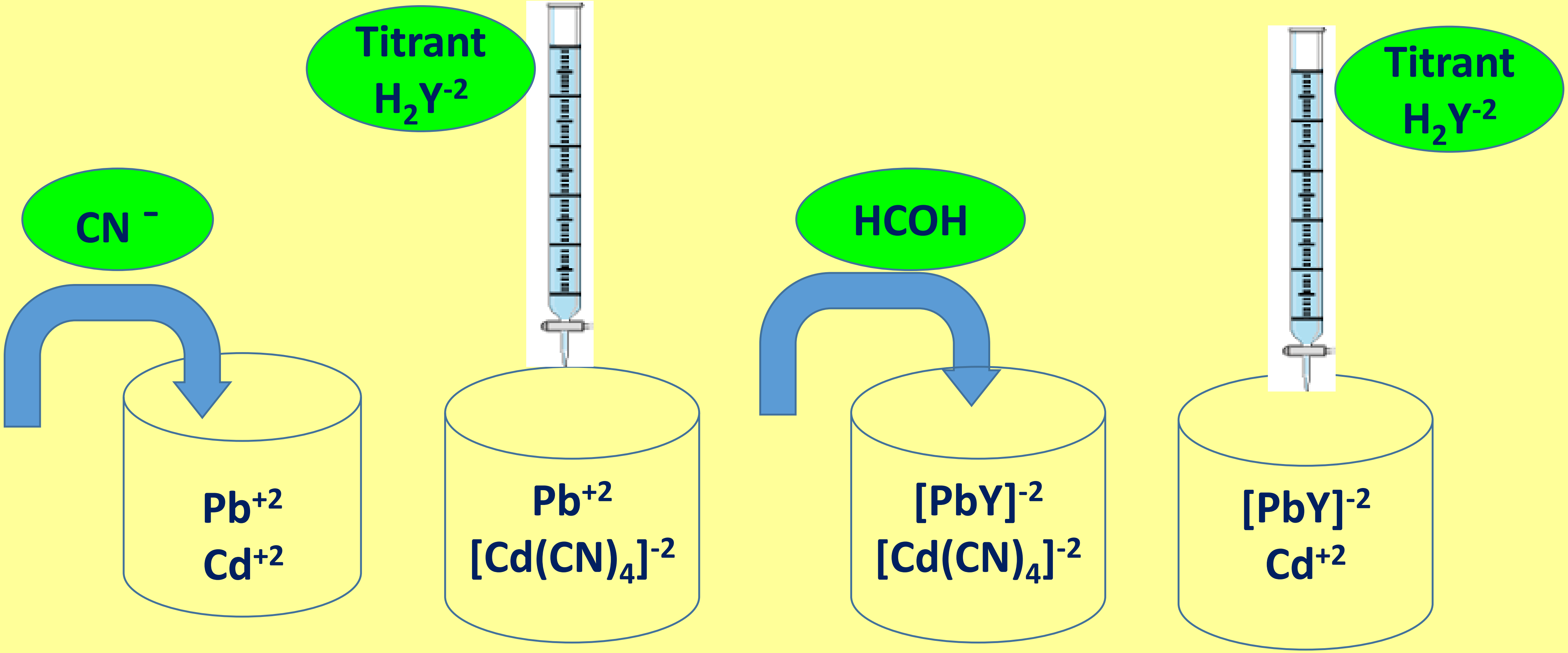
Demasking of cyanide complexes by addition of HCHO

$$[Cd(CN)_4]^{-2} + 4\ HCHO + 4\ H^{+} \longrightarrow Cd^{+2} \quad + \quad 4$$

Masking and demasking

$Pb^{+2} + H_2Y^{-2}{}_{titrant} \longrightarrow [PbY]^{-2} + 2H^{+}$ ⟵ The 1st titration

$Cd^{+2} + H_2Y^{-2}{}_{titrant} \longrightarrow [CdY]^{-2} + 2H^{+}$ ⟵ The 2ed titration



CN^{-}

Pb^{+2}
Cd^{+2}

Titrant
H_2Y^{-2}

Pb^{+2}
$[Cd(CN)_4]^{-2}$

HCOH

$[PbY]^{-2}$
$[Cd(CN)_4]^{-2}$

Titrant
H_2Y^{-2}

$[PbY]^{-2}$
Cd^{+2}

Titration Methods Involving EDTA

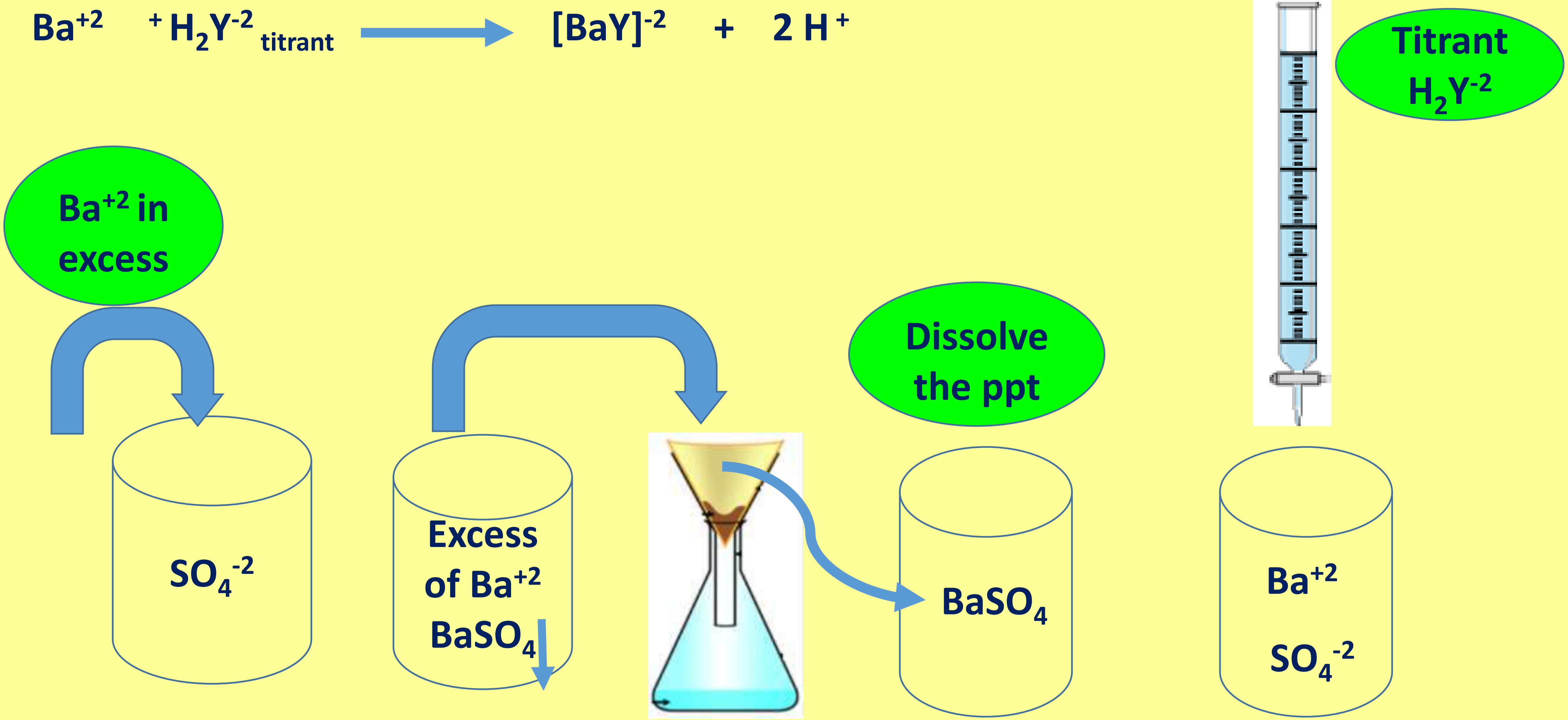
Compleximetric titration of anions

The anion is precipited as a sparingly soluble salt. The resulting precipitate is separated and redissolved. The liberated metal ion is then titrated with EDTA solution. Another procedure can be performed when an excess of metal ion is added to precipitate the anion and the rest of cation is then titrated with EDTA solution.

Complexímetric titration of anions

$Ba^{+2} + SO_4^{-2} \longrightarrow BaSO_4$

$Ba^{+2} + H_2Y^{-2}{}_{\text{titrant}} \longrightarrow [BaY]^{-2} + 2H^+$



Ba^{+2} in excess

SO$_4^{-2}$

Excess of Ba^{+2}

BaSO$_4$↓

Dissolve the ppt

BaSO$_4$

Titrant H$_2$Y^{-2}

Ba^{+2}

SO$_4^{-2}$

Complexometric Titration
Part 3

Dr. Mai Ramadan

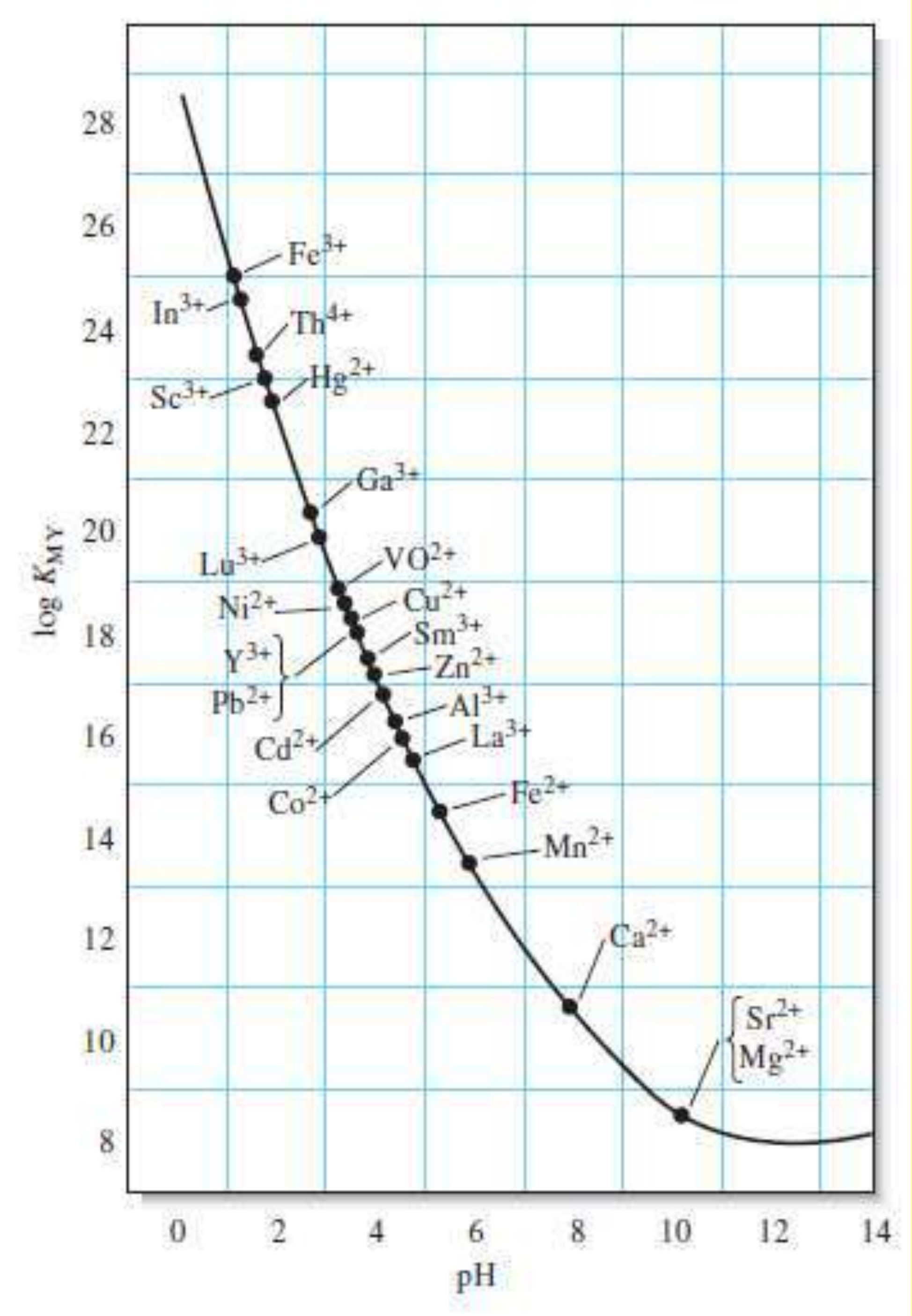
Problems

A 50.00-mL aliquot of a solution containing iron(II) and iron(III) required 10.98 mL of 0.01500 M EDTA when titrated at pH 2.0 and 23.70 mL when titrated at pH 6.0. Express the concentration of each solute in parts per million.

Solution: 184.0 ppm Fe^{+3} and 213.1 ppm Fe^{+2}

Do you remember!!!!!

Minimum pH needed for satisfactory titration of various cations with EDTA.



Problems

50.00-mL (pH=2)

No mmol EDTA = no mmol Fe^{+3} = 10.98 (mL)* 0.015 (M)=

= 0.1647 (mmol)

50 mL (pH=6)

No mmol EDTA = no mmol Fe^{+3} + Fe^{+2}= 23.7 (mL) * 0.015 (M)=

= 0.3555 (mmol)

No mmol Fe^{+2} = 0.1908 (mmol)

Conc. Fe^{+3} (ppm) = 0.1647 (mmol) *55.845 (mg/mmol) / 0.05 (L)

= **184.0 ppm**

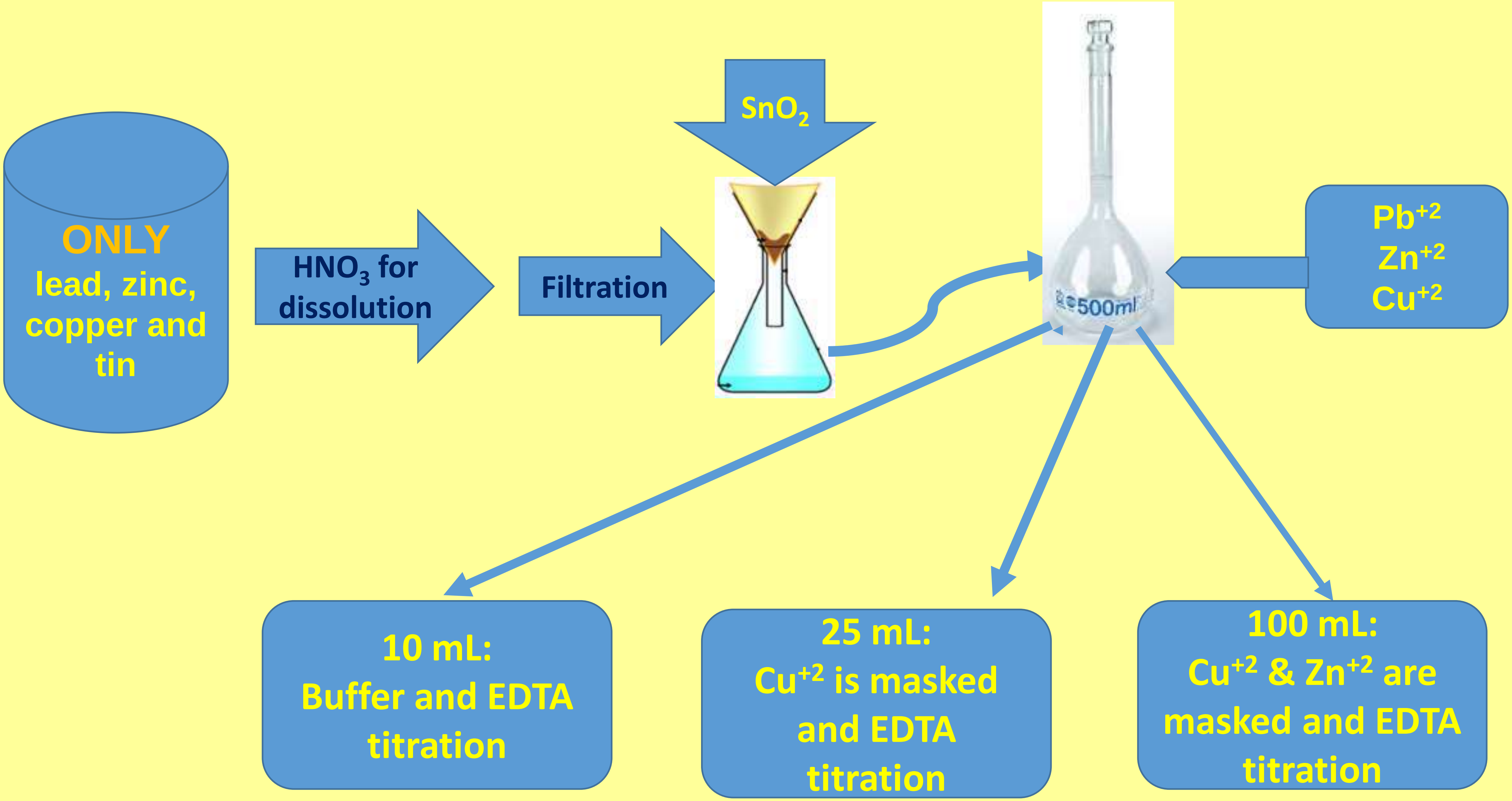
Conc. Fe+2 (ppm) = 0.1908 (mmol) * 55.845 (mg/mmol) / 0.05 (L)

= **213.1 ppm**

Problems

A 0.32 g sample contains only lead, zinc, copper and tin was dissolved in HNO3. the SnO2 precipitate was removed by filtration and the combined ions filtrate were diluted to 500 ml. A 10 ml from the solution was buffered and titrated with 37.5 ml of 0.0025 M EDTA solution. The copper in a 25 ml from the solution was masked with thiosulfate and the rest ions were titrated with 27.5 ml of the EDTA solution. Cyanide was added to mask copper and zinc in 100 ml of the solution and the rest ion was titrated with 10.5 ml of the EDTA solution. Calculate the percentage of each ion in the sample. (MW Pb = 207, Zn = 65, Cu = 63.5, Sn = 118)

Solution:



ONLY
lead, zinc, copper and tin

HNO_3 for dissolution

Filtration

SnO_2

Pb^{+2}
Zn^{+2}
Cu^{+2}

10 mL:
Buffer and EDTA titration

25 mL:
Cu^{+2} is masked and EDTA titration

100 mL:
Cu^{+2} & Zn^{+2} are masked and EDTA titration

Problems

10 ml
no mmol EDTA = 37.5 (ml)*0.0025 (M)= 0.09375 (mmol)
= no mmol(Zn + Cu + Pb) = 0.09375 (mmol)

no mmol(Zn + Cu + Pb) = 4.6875 (mmol) -------- 500 (mL)

25 ml Cu is masked
no mmol EDTA = 27.5 (mL) * 0.0025 (M)= 0.06875(mmol)
= no mmol(Zn + Pb) = 0.06875 (mmol)

no mmol(Zn + Pb) = 1.375 (mmol) -------- 500 (mL)

100 ml: Zn and Cu are masked
no mmol EDTA = no mmol Pb= 10.5 (ml)*0.0025 (M)
= 0.02625(mmol)
no mmol(Pb) = 0.13125 (mmol) -------- 500 (mL)
\

Problems

$$\% \text{ Pb} = \frac{0.13125 * 10^{-3} \text{ (mol)} * 207 \text{ (g/mol)}}{0.32 \text{ (g)}} * 100 = 8.49\%$$

no mmol(Zn in 500 mL) = 1.375 - 0.13125= 1.24375 (mmol)

$$\% \text{ Zn} = \frac{1.24375 * 10^{-3} \text{ (mol)} * 65 \text{ (g/mol)}}{0.32 \text{ (g)}} * 100 = 25.26\%$$

no mmol(Cu in 500 mL) = 4.6875 - 1.375 = 3.3125 (mmol)

$$\% \text{ Cu} = \frac{3.3125 * 10^{-3} \text{ (mol)} * 63.5 \text{ (g/mol)}}{0.32 \text{ (g)}} * 100 = 65.73\%$$

% Sn = 100% - (Pb+Cu+Zn%) = 0.52%

Problems

A sample containing lead, magnesium and zinc (0.4085 g) was dissolved and treated with cyanide to complex and mask zinc

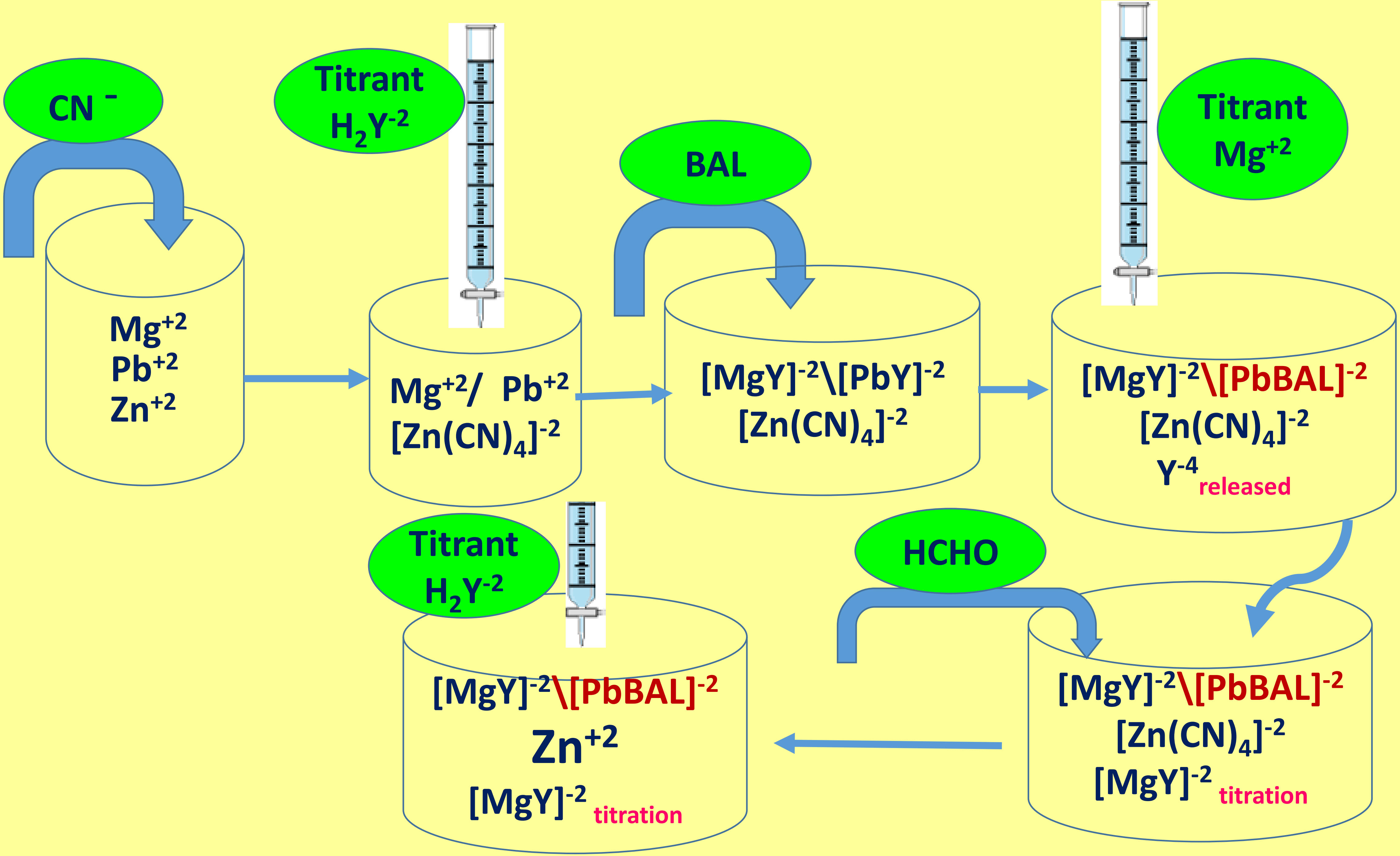
$$Zn^{+2} + 4\ CN^{-} \longrightarrow [Zn(CN)_4]^{-2}$$

Titration of lead and magnesium required 42.22 ml of 0.02064 M EDTA. The lead was next masked with BAL and the released EDTA was titrated with 19.35 ml of 0.007657 M magnesium solution. Finally the formaldehyde was introduce to demask zinc

$$[Zn(CN)_4]^{-2} + 4\ HCHO + 4\ H_2O \longrightarrow Zn^{+2} + 4\ HOCH_2CN + 4\ OH^{-}$$

which was titrated with 28.63 ml of 0.02064 M EDTA solution. Calculate the percentage of three elements in the sample.

Solution:



CN^-

Mg^{+2}
Pb^{+2}
Zn^{+2}

Titrant
H_2Y^{-2}

Mg^{+2}/ Pb^{+2}
$[Zn(CN)_4]^{-2}$

BAL

$[MgY]^{-2}\backslash[PbY]^{-2}$
$[Zn(CN)_4]^{-2}$

Titrant
Mg^{+2}

$[MgY]^{-2}\backslash[PbBAL]^{-2}$
$[Zn(CN)_4]^{-2}$
Y^{-4} released

HCHO

$[MgY]^{-2}\backslash[PbBAL]^{-2}$
$[Zn(CN)_4]^{-2}$
$[MgY]^{-2}$ titration

Titrant
H_2Y^{-2}

$[MgY]^{-2}\backslash[PbBAL]^{-2}$
Zn^{+2}
$[MgY]^{-2}$ titration

$$Pb^{+2} + H_2Y^{-2}{}_{\text{titrant}} \longrightarrow [PbY]^{-2} + 2H^{+}$$

$$Mg^{+2}{}_{\text{sample}} + H_2Y^{-2}{}_{\text{titrant}} \longrightarrow [MgY]^{-2} + 2H^{+}$$

The 1st titration

$$[PbY]^{-2} + BAL \longrightarrow [PbBAL] + Y^{-4}{}_{\text{free}}$$

$$Y^{-4}{}_{\text{free}} + Mg^{+2}{}_{\text{titrant}} \longrightarrow [Mgy]^{-2}$$

The 2ed titration

$$Zn^{+2} + H_2Y^{-2}{}_{\text{titrant}} \longrightarrow [ZnY]^{-2} + 2H^{+}$$

The 3rd titration

Problems

No mmol EDTA = no mmol (Mg + Pb) = 42.22 (mL)* 0.02064 (M)=
= 0.8714 (mmol)

$$[PbY]^{-2} + BAL \longrightarrow [PbBAL] + Y^{-4}_{free}$$

$$Y^{-4}_{free} + Mg^{+2} \longrightarrow [Mgy]^{-2}$$

No mmol Mg^{+2} (**titrant**) = no mmol $[PbY]^{-2}$ =no mmol Pb^{+2}
= 19.35 (mL) * 0.007657 (M) =0.1482 (mmol)

No mmol Mg (**Sample**) = 0.8714 - 0.1482 = 0.7232 (mmol)

No mmol EDTA = no mmol Zn^{+2} = 28.63 (ml) * 0.02064 (M) = 0.5909 (mmol)

Problems

$$\% \text{ Zn} = \frac{0.5909 * 10^{-3} \text{ (mol)} * 65 \text{ (g/mol)}}{0.4085 \text{ (g)}} * 100 = 9.40\%$$

$$\% \text{ Pb} = \frac{0.1482 * 10^{-3} \text{ (mol)} * 207 \text{ (g/mol)}}{0.4085 \text{ (g)}} * 100 = 7.51\%$$

$$\% \text{ Mg} = \frac{0.7232 * 10^{-3} \text{ (mol)} * 24.305 \text{ (g/mol)}}{0.4085 \text{ (g)}} * 100 = 4.30\%$$

Problems

Examples: 14-3
Feature 14-4, Table 14-3

Problems:
Chapter 14: 1(a-h), 2, 3, 7, 9, 10 (a, c), 11, 12, 13, 14, 15, 16, 17, 18, 19, 20, 21, 22, 23, 24

To be discussed:

14-10(a,c,e), 14-7

Problems

Why is a small amount of [MgY]-2 ofter added to a solution that is to be titrated for hardness with Eriochrome black T?
Hardness means determination of Ca+2 in water

Magnesium edetate complex is added to water the following reaction

$[MgY]^{2-} + Ca^{2+} \rightarrow Mg^{2+} + [CaY]^{2-}$

Then adjust pH to 10 and titrate Mg+2 with EDTA standard solution using Erochrom black T as indicator in this media
At pH 10 indicator color change near the equivalence point in case of Mg+2 thus titration end point determination is accurate
If you choose direct titration of Ca+2 to determine water hardness Erichrom black T color changes before the Equivalence point due to higher KM of $[CaY]^{2-}$ in comparision to $[MgY]^{2-}$ as been discussed before.

Problem

A 24-hr urine specimen was diluted to 2.0 L. after being buffered to pH 10, a 10.0 mL aliquot was titrated with 26.81 mL of 0.003474 M EDTA. The calcium in a second 10.0 mL aliquot was isolated as CaC_2O_2. Redissolved in acid and titrate with 11.63 mL of EDTA solution. Assuming that 15 – 300 mg of magnesium and 50 – 400 mg calcium per day are normal. Did this specimen fall within the range?

Solution

1. No. mmole EDTA = no. mmoleCa^{+2} + no. mmoleMg^{+2}

26.81 mL x 0.003474 M = 0.09313 mmol ………… 10 mL

18.627 mmol …………… 2000 mL

2. No. mmoleCa^{+2} = no. mmole EDTA

11.63 mL x 0.003474 M = 0.0404 mmol ………. 10 mL

8.08 mmol ……….. 2000 mL

3. No. mmoleMg^{+2} in 2000 mL = 10.547 mmol

4. Wt of Ca^{+2} in urine per day = 8.08 mmol x 40.078 = 323 mg

Wt of Mg^{+2} in urine per day = 10.547 mmol x 24.305 = 256 mg

The urine specimen fall within the normal range.

Problem

The sulfate in 1.515g sample was homogenously precipitated as $BaSO_4$ by adding Xss of $[BaY]^{2-}$ solution slowly increasing the acid concentration to liberate Ba^{+2} the precipitate was filtered and washed and the filtrate and washing were collected to 250 mL volumetric flask. At pH = 10 buffer, was added and the solution was diluted to the mark. A 25 mL aliquot required a 28.73 mL titration with 0.01545 M Mg^{+2} solution. Express the results of this analysis in terms of percent $Na_2SO_4.H_2O$

$[BaY]^{-2} \longrightarrow Ba^{+2} + Y^{-4}$ released

$Ba^{+2} + SO_4 \longrightarrow BaSO_4 \downarrow$

- ✓ Titrate 25 ml of filtrate and washings with Mg^{+2} standard solution

Y^{-4}released $+ Mg^{+2} \longrightarrow [MgY]^{-2}$

No. mmol Y^{-4}released = no. mmolMg^{+2} = 28.73 ml x 0.01545 M
= 0.444 mmol …… 25 ml
4.44 mmol ……. 250 ml

No. mmol Y^{-4} released = no. mmolBa^{+2} = no. mmolSO_4 = 4.44 mmol
No. mmol$Na_2SO_4.H_2O$ = no. mmolSO_4 = 4.44 mmol

$$\% \; Na_2SO_4.H_2O = \frac{\text{wt. } Na_2SO_4.H_2O}{\text{wt. sample}} \text{x } 100 = \frac{4.44 \text{ x } 10\text{-}3 \text{ x } 322}{1.525} \text{ x } 100 = 94.34\%$$