### Table -1 Fundamental SI Units

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**lanthanides**

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Qualitative Analysis:

analysis that gives an identification of various substance in the sample. i.e. identification of various elements that make up the sample. Ex.
To identify Ag\(^{+1}\) ion, Add Cl\(^{-1}\) ion, AgCl white precipitate indicate the presence of Ag.

Quantitative Analysis:

Deals with the determination of the amount of various substance in the sample.
Methods of Analysis :-

1- Volumetric Analysis : التحليل الحجمي
the amount of substance present is measured indirectly by determining the volume of the solution of known strength which is required to react with the sample.

Ex. Titration the processes Acid - Base titration of a solution of an acid with a solution of base used. The amount of acid is competed from the volume of base used.

2- Gravimetric Analysis: التحليل الوزني
the substance isolated as precipitant and the amount of substance is calculated from the weight of precipitant
Ex A weight of Ag+ (amount) is calculated from the weight of AgCl

3- Instrumental methods of Analysis: التحليل الاالي
instrument used to measure certain properties which are related to the amount of substance in the sample Ex 1 - spectrophotometry methods
2 - Gas chromatography
Analytical Chemistry

Reaction of the metals ions or cations

Analytical classification of metals ions:

The common metallic ions divided for the purpose of qualitative analysis into a number of groups which are distinguished by the fact that *the metals of any group are precipitated by particular group reagent*
<table>
<thead>
<tr>
<th>group</th>
<th>Group reagent</th>
<th>Ions</th>
<th>Formula of precipitate</th>
<th>Distinguished feature</th>
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<tr>
<td>I Silver group</td>
<td>Dil. HCl</td>
<td>Ag⁺, Pb⁺², Hg⁺²</td>
<td>AgCl, PbCl, HgCl₂</td>
<td>Chlorides insoluble in cold HCl (dil.)</td>
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<tr>
<td>II A copper group</td>
<td>H₂S in presence of Dil. HCl</td>
<td>Hg⁺², Pb⁺², Bi⁺³, Cu⁺², Sn⁺³, As⁺³, Sb⁺³, Sn⁺⁴</td>
<td>HgS, PbS, Bi₂S₃, CuS, As₂S₃, Sb₂S₃, SnS₃</td>
<td>Sulfides insoluble in HCl (dil.)</td>
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<td>III A Iron group</td>
<td>Aqueous NH₃ in presence NH₄Cl</td>
<td>Al⁺³, Cr⁺³, Fe⁺³</td>
<td>Al(OH)₃, Cr(OH)₃, Fe(OH)₃</td>
<td>Hydroxide Precipitated by aqueous NH₃</td>
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<td>III B Zinc group</td>
<td>H₂S in presence of Aq. NH₃ &amp; NH₄Cl</td>
<td>Ni⁺², Co⁺², Mn⁺², Zn⁺²</td>
<td>NiS, CoS, MnS, ZnS</td>
<td>Sulfides Precipitated by H₂S in presence of Aq. NH₃ &amp; NH₄Cl</td>
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<tr>
<td>IV Calcium group</td>
<td>(NH₄)₂CO₃ in presence of Aq. NH₃ &amp; NH₄Cl</td>
<td>Ba⁺², Sr⁺², Ca⁺²</td>
<td>BaCO₃, SrCO₃, CaCO₃</td>
<td>Carbonate Precipitated by (NH₄)₂CO₃ in presence of Aq. NH₃ &amp; NH₄Cl</td>
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<td>V Alkali group</td>
<td>No Particular reagent</td>
<td>Mg⁺², Na⁺¹, K⁺¹, NH₄⁺</td>
<td>Mg⁺², Na⁺¹, K⁺¹, NH₄⁺</td>
<td>Ions not precipitated in previous groups</td>
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The Silver group (group I)
(lead, Mercury(ous) and Silver)

Compounds of these elements are characterized by their precipitation as chlorides by diluted HCl

$\text{PbCl}_2$: Slightly soluble in water, is not completely precipitated as chloride in this group, it is found in group II as PbS

**Lead Pb:-**
- Bluish gray metal
- Readily dissolved in dilute HNO$_3$

$$3\text{Pb} + 8 \text{HNO}_3 \rightarrow 3\text{Pb(NO}_3\text{)}_2 + 2\text{NO} + 4\text{H}_2\text{O}$$

- With concentrated HNO$_3$, protective film of lead nitrate which is insoluble in this acid, prevent complete solute in.

- Dilute HCl and H$_2$SO$_4$ have a little (effect) or action owing to the formation of a protective film of PbCl$_2$ and PbSO$_4$
Reaction of lead ion (Pb\(^{+2}\)) :

- With dilute HCl:
  \[ \text{Pb(NO}_3\text{)}_2 + 2\text{HCl} \rightarrow \text{PbCl}_2 + 2\text{HNO}_3 \]
  White ppt.

  \[ \text{PbCl}_2 \rightarrow \text{(water Soluble in hot) Cold (separate out again in needle)} \]

- With dilute H\(_2\)SO\(_4\):
  \[ \text{Pb(NO}_3\text{)}_2 + \text{H}_2\text{SO}_4 \rightarrow \text{PbSO}_4 + 2\text{HNO}_3 \]
  White ppt.

- With potassium chromate solution:
  \[ \text{Pb(NO}_3\text{)}_2 + \text{K}_2\text{CrO}_4 \rightarrow \text{PbCrO}_4 + 2\text{KNO}_3 \]
  Yellow ppt.
Detection and Separation of group(1):
(Silver group) to a given Solution, add a diluted HCl (in excess)

Filtrate

Residue (ppt.)
AgCl, HgCl₂, PbCl₂,
Wash with little very dill. Acid

Boil with 5-10 ml of water

Filtrate

Filtrate may contain PbCl₂

Filtrate may contain other groups
Discharge
Residue may contain Hg\(_2\)Cl\(_2\) and AgCl. Ppt. is wash with hot water.

1. Add little ammonium acetate solution + K\(_2\)CrO\(_4\)

filtrate no ppt. with K\(_2\)CrO\(_4\)
(i.e.) complete removal of PbCl\(_2\).

2. Pour 3-4 ml of dil. NH\(_3\) over the ppt.

white crystalline ppt. of PbCl\(_2\) (Pb is present)

filtrate
Residue black

\[ \text{Hg}_2\text{Cl}_2 + 2\text{NH}_3 \rightarrow \text{Hg(NH}_2\text{)}\text{Cl} + \text{Hg} \]

Amino mercuric chloride

\( \text{Hg}^{+2} \) is present

Filtrate

Ag(NH\text{3})\text{2Cl} + \text{dil. HNO}_3

white ppt.

\[ \text{Ag Cl} + \text{NH}_3 \rightarrow \text{Ag(NH}_3\text{)}\text{2Cl} \]

Amino silver chloride

Ag(NH\text{3})\text{2Cl} + \text{HNO}_3

AgCl

dil.

White ppt.

\( \text{Ag}^{+1} \) is present
The Copper and Arsenic group (II)

Mercury(II), lead, Bismuth, Copper, Cadmium, Arsenic, Antimony, and Tin.

Compound of these elements are characterized by their ppt. as sulphides by $\text{H}_2\text{S}$ from 0.3N HCL

Group II

Copper group
Hg, Pb, Bi, Cu, Cd
Insoluble in (NH$_4$)S and NaOH

As, Sb, Sn
Soluble in (NH$_4$)S and NaOH
The copper Cu:

- copper is light red metal.
- Soft, malleable + ductile.
- Unaffected by dil. HCl and H₂SO₄.

Cu + 2H₂SO₄ → CuSO₄ + SO₂ + 2H₂O
warm, conc.

- Readily attracted by HNO₃

3Cu + 8 HNO₃ → 3Cu(NO₃)₂ + 4H₂O + 2NO
dil.
Reaction of the cupric ion Cu$^{+2}$

1- With $\text{H}_2\text{S}$:

$$\text{CuSO}_4 + \text{H}_2\text{S} \rightarrow \text{CuS} + \text{H}_2\text{SO}_4$$
black ppt.

$$3\text{CuS} + 8 \text{HNO}_3 \rightarrow 3\text{Cu(NO}_3)_2 + 4\text{H}_2\text{O} + 2\text{NO} + 3\text{S}$$
Hot dil.

2- With NaOH:

$$\text{CuSO}_4 + \text{NaOH} \rightarrow \text{Cu(OH)}_2 + \text{Na}_2\text{SO}_4$$
Cupric hydroxide blue ppt.

$$\text{Cu(OH)}_2 \rightarrow \text{CuO} + \text{H}_2\text{O}$$
Blue ppt.

3- With potassium Ferrocyanide:

$$3\text{CuSO}_4 + \text{K}_4[\text{Fe(CN)}_6] \rightarrow \text{Cu}_2[\text{Fe(CN)}_6] + 2\text{K}_2\text{SO}_4$$
Reddish - brown ppt.
The Tin (Sn) :-

- Tin : silver white metal.
- Malleable and ductile at ordinary temperature.
- Metal dissolves slowly in dilute HCl and dilute H₂SO₄ with the liberation of H₂ and formation of stannous salts.

\[
\text{Sn} + 2\text{HCl} \rightarrow \text{SnCl}_2 + \text{H}_2 \\
\text{Sn} + \text{H}_2\text{SO}_4 \rightarrow \text{SnSO}_4 + \text{H}_2
\]
Readily dissolved by hot concentrated acids

\[ \text{Sn} + \text{HCl} \rightarrow \text{SnCl}_2 + \text{H}_2 \]

Conc.

\[ \text{Sn} + 4\text{H}_2\text{SO}_4 \rightarrow \text{Sn(SO}_4)_2 + 2\text{SO}_2 + 4\text{H}_2\text{O} \]

Concentrated

\[ 4\text{Sn} + 10\text{HNO}_3 \text{ (dill)} \rightarrow 4\text{Sn(NO}_3)_2 + \text{NH}_4\text{NO}_3 + \text{H}_2\text{O} \]

Dissolve slowly

*No gas in last reaction*
Reactions of stannous compound

With $\text{H}_2\text{S}$:

$$\text{SnCl}_2 + \text{H}_2\text{S} \rightarrow \text{SnS} + 2\text{HCL}$$

Brown ppt.
Stannous sulphides

With $\text{NaOH}$

$$\text{SnCl}_2 + 2\text{NaOH} \rightarrow \text{Sn(OH)}_2 + 2\text{NaCl}$$

White ppt.
Stannous hydroxide

With $\text{HgCl}_2$ mercuric chloride:

$$\text{SnCl}_2 + 2\text{HgCl}_2 \rightarrow \text{SnCl}_4 + \text{Hg}_2\text{Cl}_2$$

White ppt.
Mercurous chloride
The Iron and Zinc group (group III)
Iron, aluminum, chromium, nickel, cobalt, manganese, and zinc. All ppt. in presence of NH$_4$Cl

Group(III)A (Iron) :-
Fe, Al, Cr ppt. as Hydroxide by NH$_3$ Sol.
+ NH$_4$

Group(III)B (Zinc)
Ni, Co, Mn, Zn remain in Sol. And may be ppt by H$_2$S as sulphides

Iron group: Iron (Fe):-
_ Pure iron: is silver white.
_ Ductile and tenacious metal.
\[- \text{Fe} + 2\text{HCl} \rightarrow \text{FeCl}_2 + \text{H}_2 \]

Dill and conic

\[- \text{Fe} + \text{H}_2\text{SO}_4 \rightarrow \text{FeSO}_4 + \text{H}_2 \text{ dissolve with H}_2 \]

\[2\text{Fe} + 6\text{H}_2\text{SO}_4 \ (\text{hot and conc.}) \rightarrow \text{Fe}_2(\text{SO}_4)_3 + 6\text{H}_2\text{O} + 3\text{SO}_2 \]

\[4\text{Fe} + 10\text{HNO}_3 \ (\text{cold and dill}) \rightarrow 4\text{Fe(NO}_3)_2 + \text{NH}_4\text{NO}_3 + 3\text{H}_2\text{O} \]

\[4\text{Fe} + 10\text{HNO}_3 \ (\text{warm and dill}) \rightarrow 4\text{Fe(NO}_3)_2 + \text{N}_2\text{O} + 5\text{H}_2\text{O} \]
Volumetric Analysis (Titrimetric Analysis)

**Calculation involving concentration of solution**

Measurements usually consist of a unit and a number expressing the quantity of that unit. Many different units may be used to express the same physical measurement. For example, the mass of a sample weighing 1.5 g also may be expressed as 0.0033 lb.

**Concentration** is a general measurement unit stating the amount of solute present in a known amount of solution

\[
\text{Concentration} = \frac{\text{amount of solute}}{\text{amount of solution}}
\]

Although the terms “solute” and “solution” are often associated with liquid samples they can be extended to gas-phase and solid-phase samples as well.

**Solution**: - Homogenous mixture of one or more components in one phase.
The concentration of solution is expressed by:

1- Physical methods

2 - Chemical methods

   Physical methods:-

   Gram of solute / liter or (100 ml) of solution

1. Gram of solute / liter or (100 ml) of solvent
2. Gram of solute / 1000 gm or (1 Kg) or (100 gm) of solution
3. Gram of solute / 1000 gm or (1 Kg) or (100 gm) of solvent

1. Percentage methods:

   a - Weight percent (% wt) = \( \frac{\text{weight of solute}}{\text{weight of solution}} \times 100 \)

   b - Volume percent (% vol) = \( \frac{\text{volume of solute}}{\text{volume of solution}} \times 100 \)

   c - Weight / volume percent (%) = \( \frac{\text{weight of solute}}{\text{volume of solution}} \times 100 \)
Chemical Methods:-

Molar Solution :- a solution that contains 1 mole of solute per liter of solution
1- Molarity : number of moles of solute per liter of solution

\[ M = \frac{\text{no. of moles}}{\text{liter of solution}} \]
No. of moles = M x Liter of solution

2- Molal Solution:- A solution that contains one mole of solute per 1 kilograms of solvent.

\[ m = \frac{\text{no. of mole of solute}}{1 \text{ Kg of solvent}} \]
3-**Formal Solution**:–
A solution that contain one formula weight of solute per liter of solution.

\[ F = \frac{\text{weight}}{\text{Fwt}} \times \frac{1}{\text{liter of solvent}} \]

4-**Equivalent methods**

**A) Equivalent weight of atoms of element:**

\[ \text{Eq. wt} = \frac{\text{atomic weight}}{\text{valency}} \]

**A) Equivalent weight of compounds:**

1. **Equivalent weight of Acids:**
   Equivalent weight of acid which contains one replaceable hydrogen

\[ \text{Eq. wt of acid} = \frac{\text{molecular weight}}{\text{no. of hydrogen atom}} \]
2 Equivalent weight of a base;

Equivalent weight of base which contains one replaceable hydroxyl group

\[
\text{Eq. wt of a base} = \frac{\text{molecular weight}}{\text{no. of hydroxyl groups}}
\]

3 Equivalent weight of a salt;

Equivalent weight of a salt in grams which contains one weight of cat ion can react or replaced by one gram of hydrogen

4 Equivalent weight of Reducing and Oxidizing agent

\[
\text{Eq. wt. of reducing agent} = \frac{\text{molecular weight}}{\text{no. of electrons losses in the reaction}}
\]

\[
\text{Eq. wt of oxidizing agent} = \frac{\text{molecular weight}}{\text{no. of electrons gains in the reaction}}
\]
5 Normal solution;
   A solution that is contains one equivalent of solute per liter solution.

Normality= (no. of equivalent of solute )/(liter of solution)

Normality=(wt.)/(eq.wt.) X(1 )/(liter of solution)

**Part per million (PPM):**

\[ \text{ppm} = \frac{\text{mili grams of solute}}{\text{liter of solution}} \]

millequivalents and mllimoles:
It is convenient to select the unite of concentration by the amount of solute per milliliter of solution; the molar solution is then defined as one containing 0.001 moles per milliliter of solution. Likewise a normal solution is one that contains 0.001 equivalent, or millequivalents per liter
Dilution of solution:
A solution of known normality is frequently prepared from a more concentrated one of known strength by quantitative dilution.

\[(\text{Normality} \times \text{volume})_{\text{conc}} = (\text{Normality} \times \text{volume})_{\text{dilute}}\]

\[(N \times V)_{\text{conc}} = (N \times V)_{\text{dil}}\]

Primary standard:
In volumetric analysis the usual practice is to prepare solutions of approximate desired strength and then determine the exact strength by titration against a solid primary standard. The primary standard must be:
1. Of high purity 99.99%
2. Of high equivalent weight
3. Stability on drying
4. Of definite chemical formula
5. Has no affinity for water vapor or CO\text{2}
Type of the chemical in titration process:

1. Neutralization methods
   - Neutralization of $H^+$ with $OH^-$ $\rightarrow$ $H_2O$
   - Widely used as basis and volumetric determination of acids, bases, and salt of weak acids
   - The reaction is characterized by a rapid change in pH near the equivalence point.
   - The change in pH is detected by the:
     a. Acid base indicator
     b. By followed electrically pH-meter

Neutralization method:-
Acidimetry, acid is standard
Alkalimetry, base is standard
2 Oxidation – Reduction methods

Reaction in which there is change in oxidation state of two substances in the reaction.

ex.

\[ \text{Ce}^{4+} + \text{Fe}^{2+} = \text{Ce}^{3+} + \text{Fe}^{3+} \]

The two reaction occur simultaneously in general:

\[ \text{KMnO}_4 = \text{strong oxidizing agent} \]
\[ \text{H}_3\text{AsO}_3 = \text{strong reducing agent} \]
\[ \text{Na}_2\text{S}_2\text{O}_3 = \text{specific reagent for I}_2 \]

3 Precipitation Titrations

There is change in the concentration of the ions of the precipitate at the equivalence point.

*the conditions are usually adjusted that only one precipitation is formed at the equivalence point.

4 Complex ion titration

Depend on the formation of complex between the sample and titrating reagent

Ex. Determination of hardness of water i.e. determination of Mg and Ca ions in water.
Calculation in titrimetric analysis
a) calculation based on normality of solution
b) calculation based on molarity of solution:

Back titration:
To calculate a substance like (B)  \( A + B \rightarrow \text{product} + \text{Excess A} \)
Excess A +C \( \rightarrow \) Product

Back titration term of normality
\[
\text{meq} B = [(N_A \times V_A) - (V_C \times N_C)] \text{ eq. wt}_B
\]

Back titration term of molarity
\[
aA + bB = \text{product} + \text{excess } A \quad R_1 = \frac{b}{a} \frac{\text{unknown}}{\text{known}}
\]
\[
cC + dA(\text{excess}) = \text{product} \quad R_2 = \frac{d}{c}
\]
\[
m \text{ g} B = [(M_A \times V_A) - (V_C \times M_C \times R_2)] \text{ M. wt}_B
\]
Equilibrium constant

Chemical reaction at equilibrium when Forward reaction = backward reaction

For the relation

\[ aA + bB = cC + dD \]

\[ k_e = \frac{[C]^c.[D]^d}{[A]^a.[B]^b} \]

\( k_e \) = equilibrium constant

\([c],[D]\)=moles conc. of products.

\([A],[B]\)= moles conc. of reactant

A,b,c,d, coefficient of A,B,C,D

Ionization of acid:-

Ionization constant of weak electrolytes:-

- Salt of strong acid and strong Base :-

Since neither ion can combine with an ion of water to form a weak electrolyte
the pH is 7.0

PH of solution of weak electrolytes
• **Weak Acid**: (In absence of its salts) :-
• **Weak Base**: (in absence of its salts) :-
• Weak acid plus its salt
• Weak base plus its salt
• Salt of weak acid and strong base
• Salt of weak base and strong acid
Titration curves: -

- graphs of PH versus volume of reagent added in titration are known as titration curves.

PH may be obtained by:

1-Calculation
2-PH-meter

- **Titration curve of strong acid and strong base**

- **Titration curve of strong base at weak acid**
Acid-Base Indicators:-
Acid-Base Indicators are highly colored organic dyes which exhibit change in color when the PH of the solution changes between certain limits.

Indicators:

Mono colored
- e.g. ph.ph
- Phenolphthalein

Di colored
- M.O
- Methyl organic
Equilibrium in precipitation reaction

- Solubility product: -
- Evolution of $k_{sp}$ value from experimental measurements
- Computation of solubility from $k_{sp}$
- Effect of common ion on solubility
- Titration curve for precipitation reaction
Quantitative gravimetric analysis:

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depends on the actual weight of the substance or its derivatives methods:

- Precipitation methods
- Volatilization methods

Gravimetric Factor

\[ G.F = \frac{\text{equivalent weight of unknown substance}}{\text{equivalent weight of known substance}} \]

\[ \% \text{ substance A} = \frac{G.F \times \text{wt.of substance B}}{\text{wt.of sample}} \times 100 \]