Electroanalytical methods

Electroanalytical methods

- Electrogravimetry
- Coulometry
- Potentiometry
- Voltammetry

Potentiometry

- Fundamentals of potentiometry
- Reference electrodes
- Indicator and ion selective electrodes
- Instrumentation and measurement of cell electromotive force (e.m.f)

Fundamentals of potentiometry

When a metal is immersed in a solution containing its own ions, the potential difference is established between the metal and the solution

$$\mathbf{Ox + ne \rightleftharpoons Red}$$

$$\varphi = \varphi^{\theta} + \frac{RT}{nF}\ln\frac{a_0}{a_R}$$

Fundamentals of potentiometry

$$M^{n+} + ne = M$$

$$\varphi = \varphi^{\circ} + (RT/nF)\ \ln \alpha_{M^{n+}}$$

Nernst equation

Fundamentals of potentiometry

Indicator electrode

+

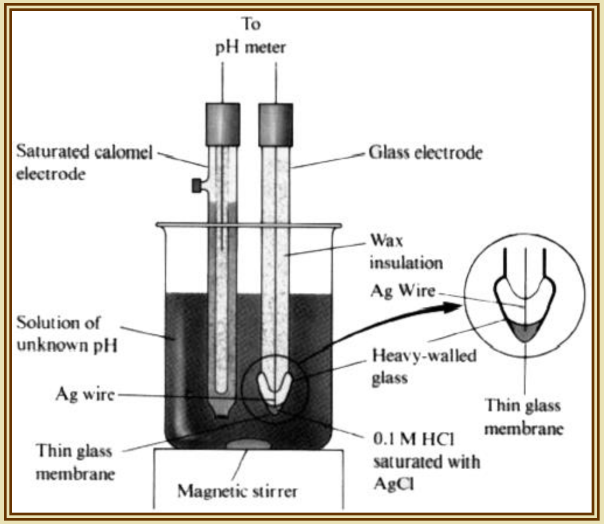
Reference electrode

+

Solution

↓

Cell



Fundamentals of potentiometry

M | M $^{n+}$ || reference electrode

$$E = \varphi_{(+)} - \varphi_{(-)} + \varphi_{L}$$

Liquid junction potential

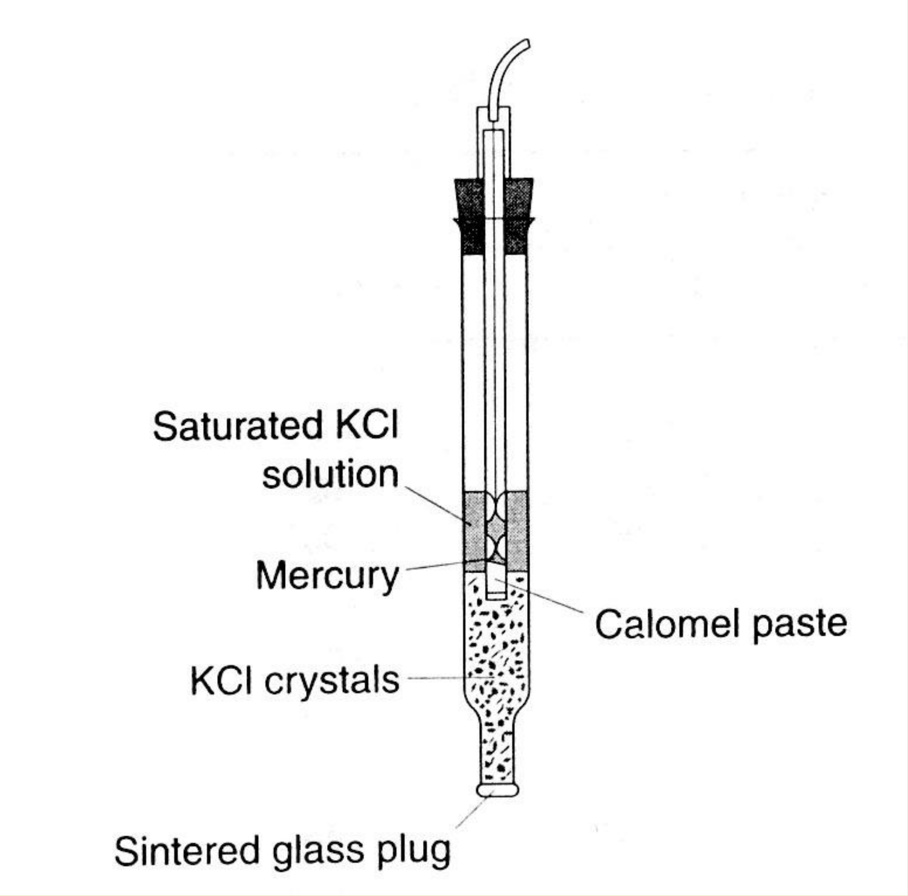
$$E = \varphi_{(+)} - \varphi_{(-)}$$

$$= \varphi_{r} - \varphi^{\circ} - (RT/nF) \ln \alpha_{Mn+}$$

Reference electrodes

- Hydrogen electrode
- Calomel electrode
- Silver – silver chloride electrode

Calomel electrode



Calomel electrode

$Hg \mid Hg_2Cl_2, \; KCl(xM) \parallel$

$$Hg_2Cl_2(s) + 2e \rightleftharpoons 2Hg(l) + 2Cl^-$$

Electrode potential

?

$$\varphi = \varphi^\circ_{Hg_2Cl_2/Hg} + (RT/nF)\ln(1/\alpha_{Cl}^{-2})$$

$$= \varphi^\circ_{Hg_2Cl_2/Hg} - 0.059 \lg \alpha_{Cl^-}$$

Silver – silver chloride electrode

Ag│AgCl，KCl(xM)‖

$AgCl(s) + e \rightleftharpoons Ag(s) + Cl^-$

Electrode potential

$$\varphi = \varphi^{\circ}_{AgCl/Ag} + (RT/nF)\ln(1/\alpha_{Cl^-})$$

$$= \varphi^{\circ}_{AgCl/Ag} - 0.059\lg\alpha_{Cl^-}$$

Indicator and ion selective electrodes

Indicator electrode

---The potential depends on the activity of a particular ionic species which it is desired to quantify

Indicator and ion selective electrodes

- Electrode of the first kind
- Electrode of the second kind
- Inert electrode

} **Metal electrode**

****

- The glass electrode
- Crystalline membrane electrode
- Biochemical electrode

} **Membrane electrode**

Indicator and ion selective electrodes

Electrode of the first kind

---The ion to be determined is directly involved in the electrode reaction

Metal M immersed in a solution of **M^{n+} ion**

$$\varphi = \varphi^{\circ}_{M^{n+}/M} + (RT/nF) \ln \alpha_{M^{n+}}$$

Indicator and ion selective electrodes

Electrode of the second kind

Silver – silver chloride electrode

--- coating a silver wire with silver choloride

$$AgCl(s) + e \rightleftharpoons Ag(s) + Cl^-$$

$$\varphi = \varphi^{\circ}_{AgCl/Ag} + (RT/nF)\ \ln (1/\alpha_{Cl^-})$$

$$= \varphi^{\circ}_{AgCl/Ag} - 0.059\ \lg \alpha_{Cl^-}$$

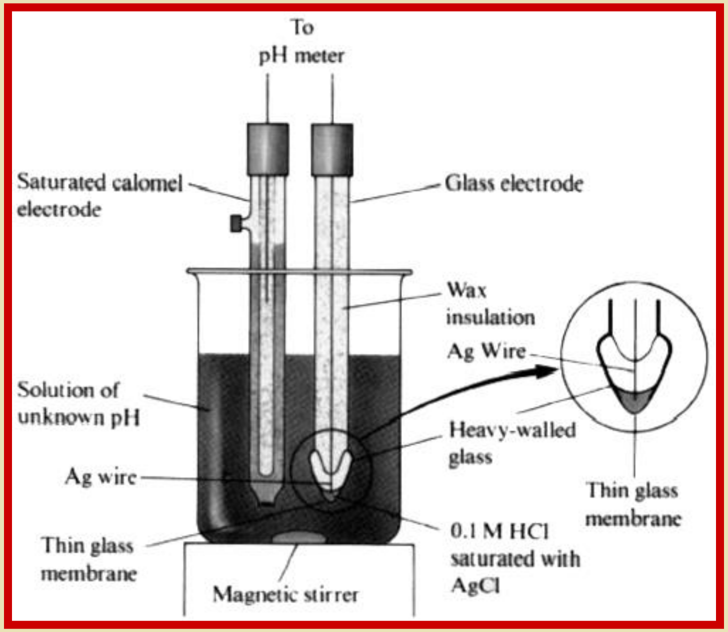
Inert electrode

---An inert electrode (Pt) is place in a system containing both an oxidizing agent and its reduction product

$$Fe^{3+} + e \rightleftharpoons Fe^{2+}$$

$$\varphi = \varphi^{\circ}_{Fe\ 3+ / Fe2+} + (RT/nF)\ \ln\ (\alpha_{Fe}{}^{3+} / \alpha_{Fe}{}^{2+})$$

The glass electrode



The glass electrode

Composition

- SiO_2 72% + Na_2O 22% + CaO 6%
- SiO_2 63% + Li_2O 28% + Cs_2O 2% + BaO 4% + La_2O_3 3%

The glass electrode

Theory

--- Ion exchange process

$$\varphi_{glass} = K + (RT/nF) \ln \alpha_{H^+}$$

$$= K' - 0.059\ pH$$

The glass electrode

properties

- Can be used in the presence of strong oxidants and reductants
- Can be used in viscous media
- Can be used in the presence of proteins

The glass electrode

properties

- High resistance
- Acid error and alkaline error

Crystalline membrane electrode

composition

Crystal of lanthanum fluoride

+

0.1 mol/L NaF – 0.1 mol/L NaCl

+

Silver – silver chloride electrode

↓

Lanthanum fluoride eletrode

Crystalline membrane electrode

Theory

Lattice defect

$$\varphi_{membrane} = K - (RT/nF)\ \ln \alpha_{F^-}$$

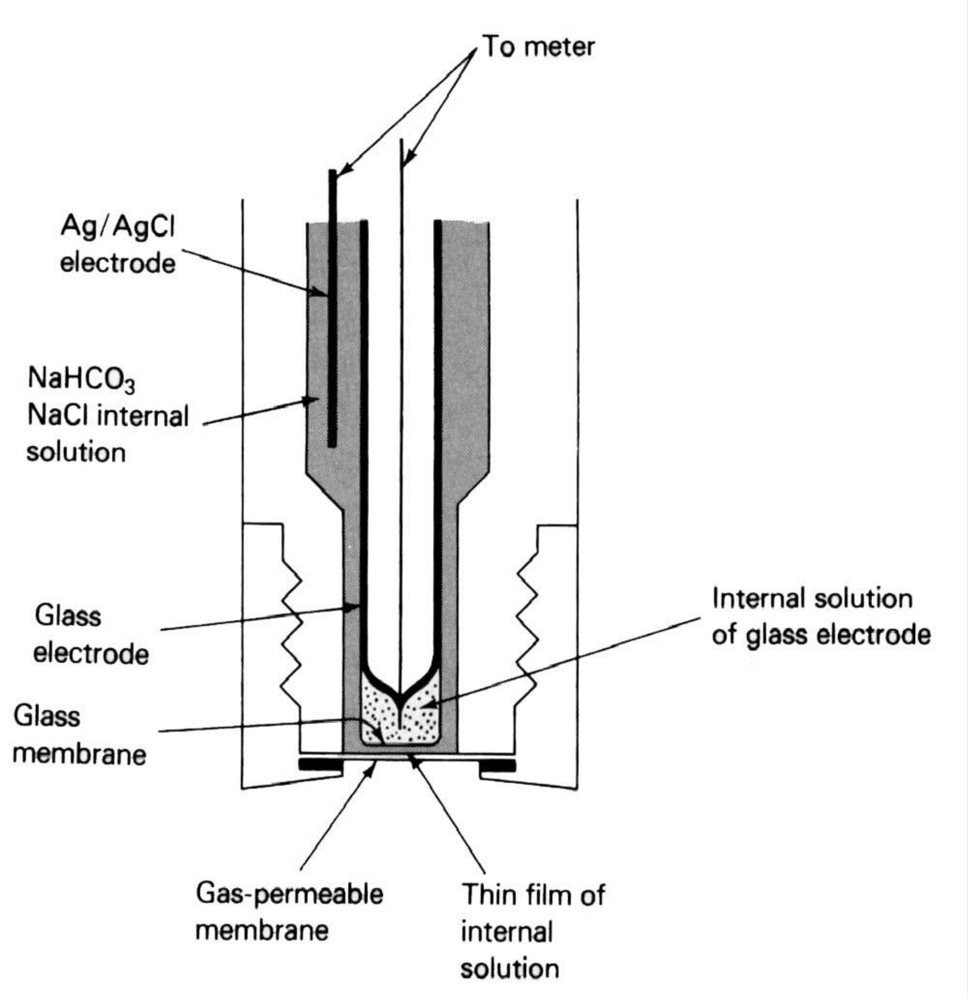
$$= K - 0.059\ \lg \alpha_{F^-}$$

Crystalline membrane electrode

properties

- Detection limit ~ 10^{-7} mol/L
- Interference ~ OH^-
- pH range ~ 5 - 6

Gas – sensing electrode



NH_3 – NH_4Cl

CO_2 – $NaHCO_3$

NO_2 –$NaNO_2$

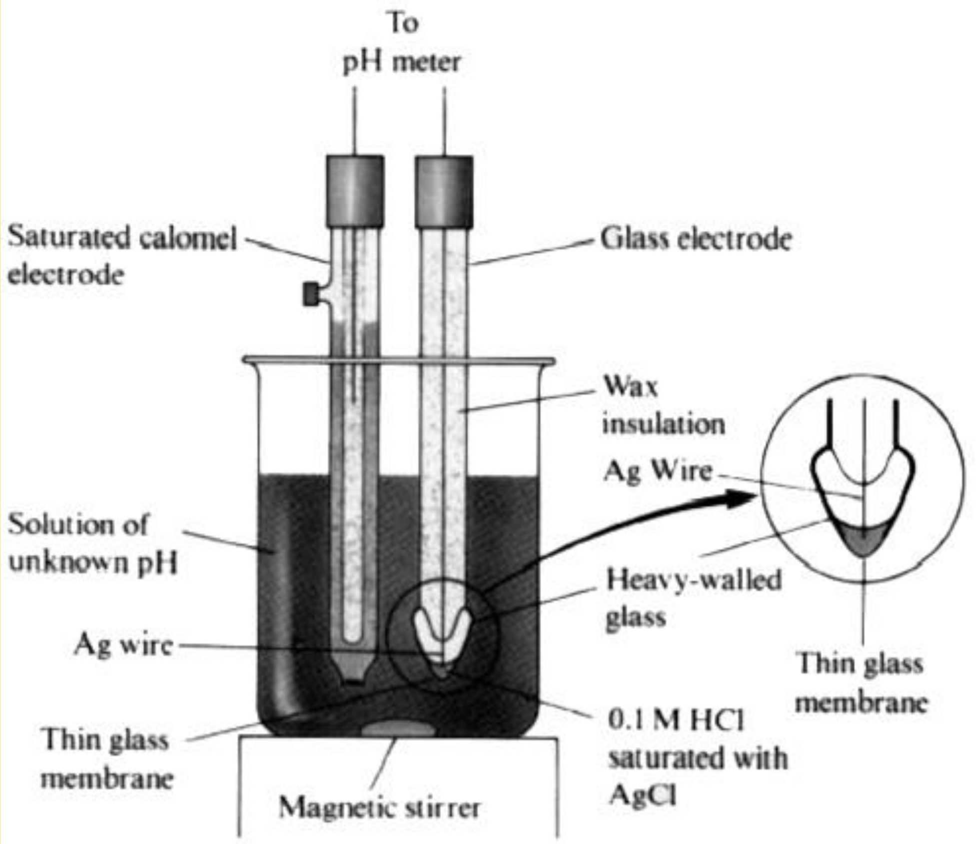
Biochemical electrode

Urea electrode

$$CO(NH_2)_2 + H_2O + 2H^+ \xrightarrow{\text{urease}} 2NH_4^+ + CO_2$$

Instrumentation

Determination of pH



Determination of pH

Glass electrode | Solution X || SCE

$$E = \varphi_{SCE} - \varphi_{glass}$$

$$= \varphi_{SCE} - (\varphi^{\circ}_{AgCl/Ag} + K + (RT/nF) \ln \alpha_{H+})$$

$$E = K' + (2.303\ RT/F)\ pH$$

Determination of pH

$$E_x = K'_x + (2.303\ RT/F)\ pH_x$$

$$E_s = K'_s + (2.303\ RT/F)\ pH_s$$

$$K'_x = K'_s$$

$$pH_x = pH_s + (E_x - E_s)\ F/2.303RT$$

--- **Operational definition**

Determination of pH

pH standard solution (25 C°)

Solution	0.05 M potassium hydrogenphthalate	0.025 M KH_2PO_4 0.025 M Na_2HPO_4	0.01 M Borax
pH	4.004	6.864	9.182

Determination of fluoride

$$\varphi_{\text{membrane}} = K \pm (0.059/n) \lg \alpha$$

- Calibration curve
- Standard addition

Determination of fluoride

Calibration curve

- Standard solutions
- Total ionic strength adjustment buffer(TISAB)
  - NaCl — Ionic strength
  - NaAc - HAc — pH
  - Sodium citrate — Interference

Determination of fluoride

Standard addition

$$E_1 = k_c + k \log y_1 C_1$$

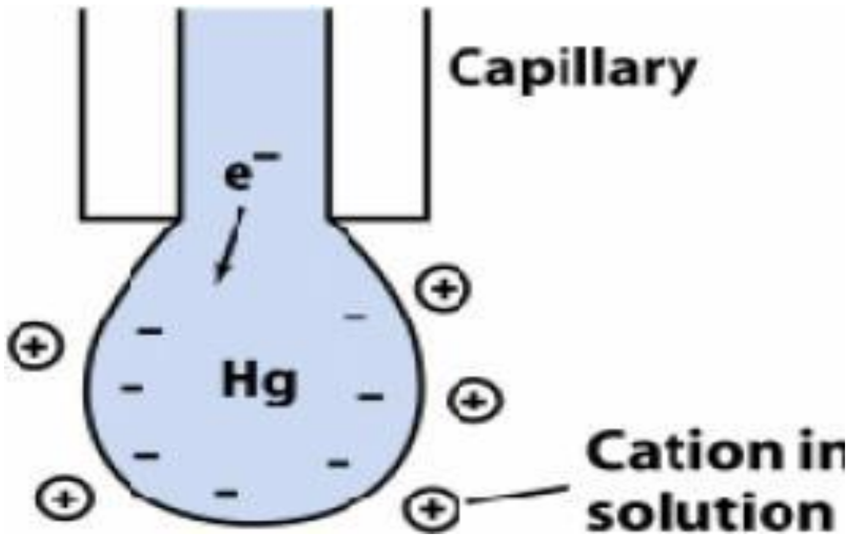
$$E_2 = k_c + k \log y_1 (V_1C_1 + V_2C_s)/(V_1 + V_2)$$

$$E_2 - E_1 = k \log (V_1C_1 + V_2C_s)/ C_1(V_1 + V_2)$$

$$C_1 = C_s/(10^{\Delta E/k}(1+V_1/V_2) – V_1/V_2)$$

Polarography

Dropping Mercury Electrode (DME)



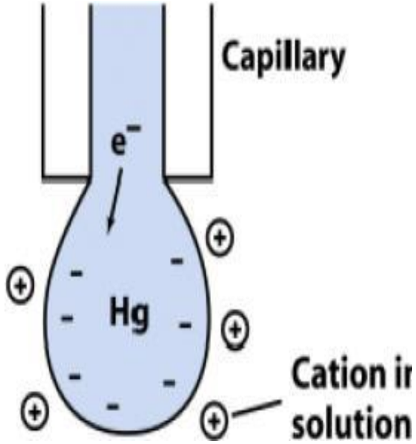
Polarography

- ❑ Polarography is one of the Voltametric methods of analysis;
electrochemical methods where current voltage curves obtained at the
surface of microelectrodes are studied.
- ❑In polarography the microelectrode is a dropping mercury
electrode (DME).
- ❑The method is used for the analysis of electroreducible or oxidizable
metal, ion or organic substance (electroactive species).
- ❑

Electroactive species is transferred into a polarographic cell
(electrolytic cell) where voltage is applied to the electrodes

- One of the electrodes is a polarizable microelectrode (DME) while the other is reference non polarizable electrode

- -DME is the cathode
- (attached to the negative
- pole of the voltage supply)
- -upon applying the voltage,
- electroactive species will
- move towards DME,
- electron transfer occurs and
- a current flows.
- -The current produced is
- proportional to
- concentration of the
- electroactive species



Polarization:

Ohm's law : $E_{cell} = I R$

E □ I (current)

If the increase in cell potential is not accompanied by increase in current it is called Polarization.

Modes of Transport of Electroactive species to DME

1-By Convection:

by mechanical stirring or by heating, as it increases current increases . This type can be prevented by:

-avoiding stirring

-controlling the temperature.

-adding gelatin to increase viscosity of medium

2-By electrostatic attraction :

between positive species and the negative cathode; The current produced here is known by migration current, it can be minimized by:

-adding large excess of inert electrolyte (not reducible) known by supporting electrolyte (50- 100 time analyte concentration)

3-By diffusion:

-occurs due to concentration gradient of ions

-The rate of mass transport by diffusion depends on theconcentration and the diffusion coefficient (a constant
value characteristic for the analyte)

-the transport (current) will depend on concentration.
Small ion conc small inflection of curve
high ion conc large inflection of curve
in polarographic analysis the mode of mass transport should be only by diffusion.

Instrument (Polarograph):

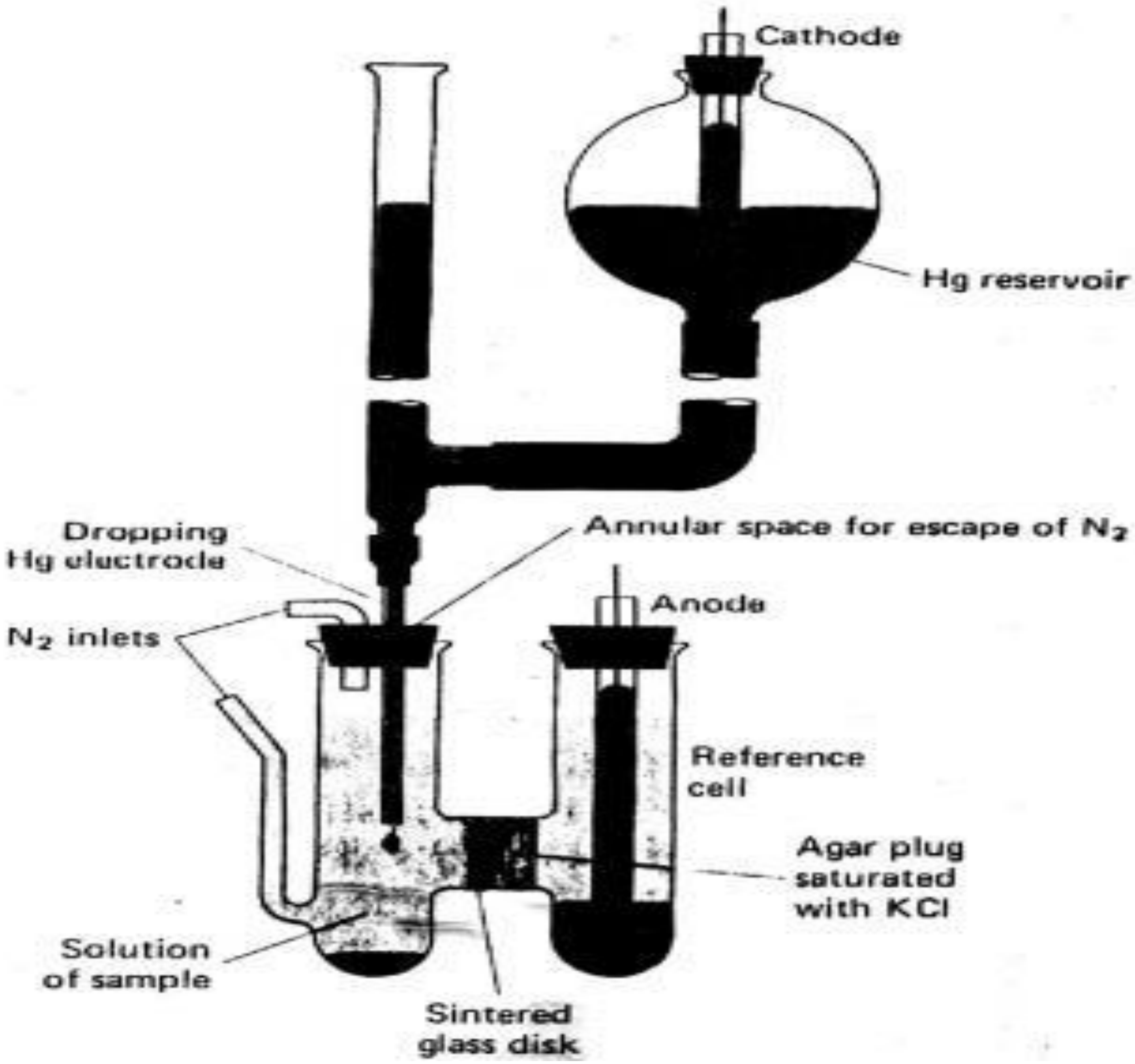
- 1-electric circuit
- 2-polarographic cell

1-electric circuit

- -increasingly negative potential from +0.5 to -2.5 volt at a definite rate of millivolt.

2-Polarographic cell

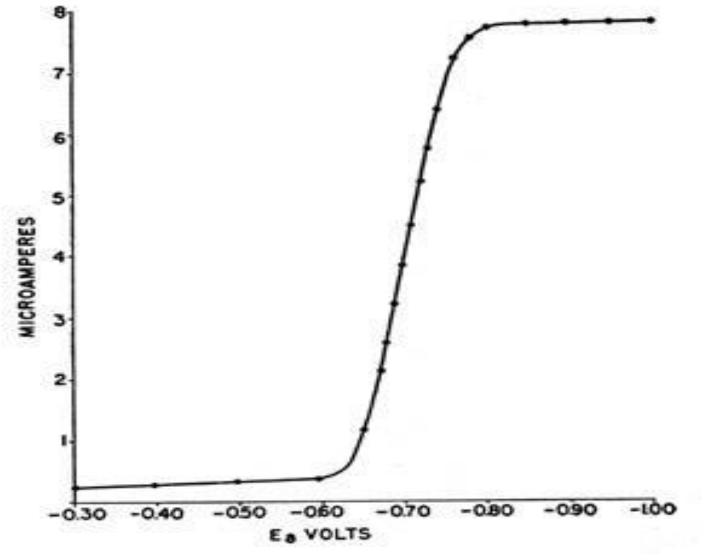
- -lifetime of a drop from 2 to 6 seconds
- - Nitrogen is bubbled through the solution for five minutes to expel oxygen. Also kept at the surface

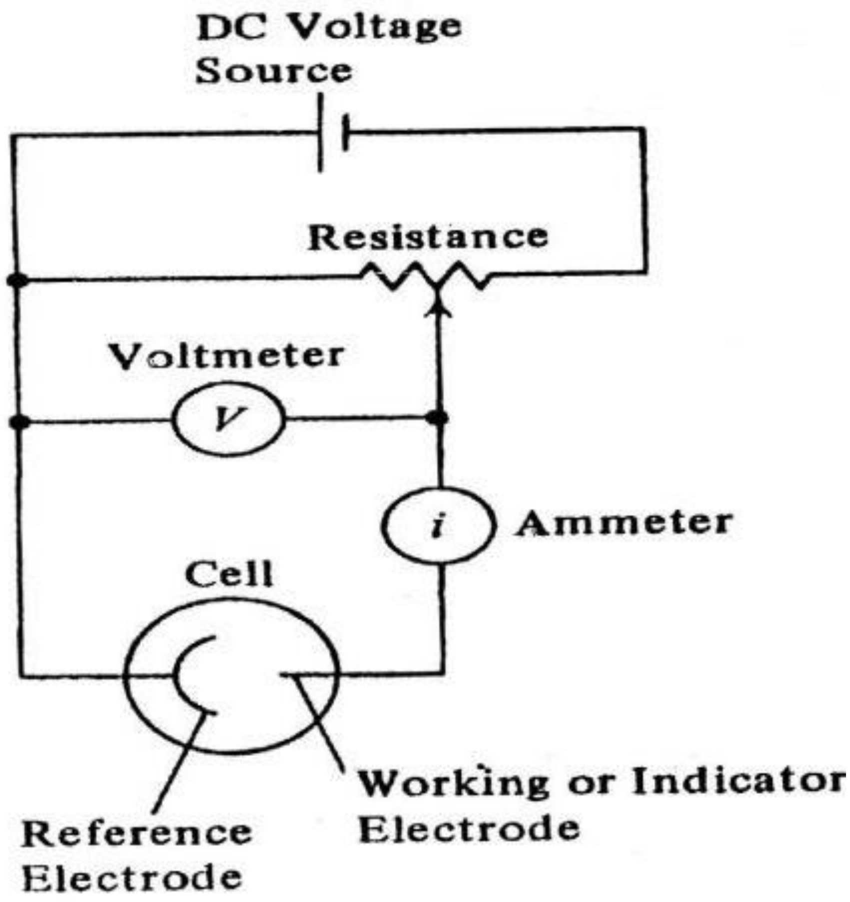

Cathode
Hg reservoir
Dropping
Hg electrode
Annular space for escape of N_2
Anode
N_2 inlets
Reference
cell
Agar plug
saturated
with KCl
Solution
of sample
Sintered
glass disk

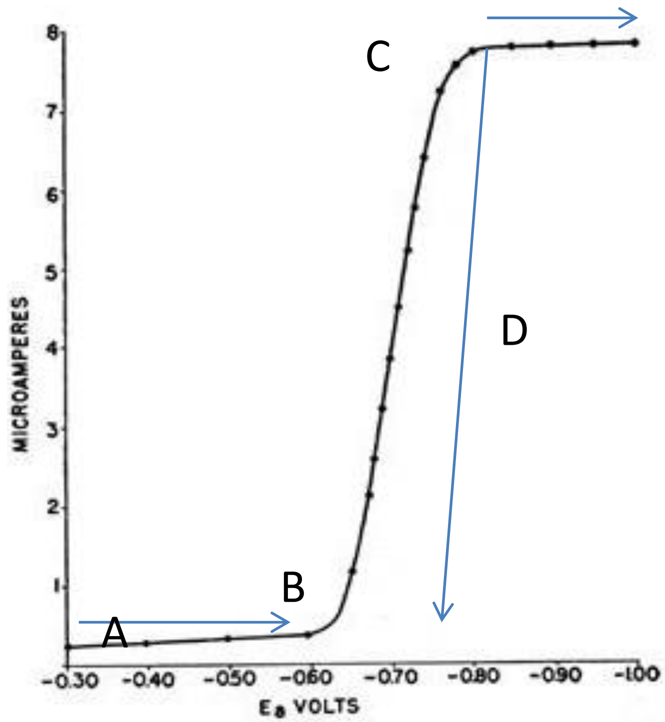
Polarogram •

I in •

-a plot of current as a •
function of applied •
potential •
-The applied •
potential is given a •
negative sign as the •
microelectrode is •
connected to the •
negative terminal of •
the power supply. •




DC Voltage
Source
Resistance
Voltmeter
V
i
Ammeter
Cell
Working or Indicator
Electrode
Reference
Electrode



8
7
6
5
4
3
2
1
MICROAMPERES
C
D
B
A
-0.30
-0.40
-0.50
-0.60
-0.70
-0.80
-0.90
-1.00
Ea VOLTS

A-B : Activation polarization
Increase in volt not accompanied by increase incurrent , additional potential is needed to overcome the energy Barrier. **B : Decomposition potential**
Potential once exceeded, reduction begins
B-C : **Increase in volt is accompanied by increase in current**
(diffusion current)
small current passing through the cell is known by residual current i_r

Ilkovič Equation:

$I_d = 607\, n\, D^{1/2}\, C\, m^{2/3}\, t^{1/6}$

Id average diffusion current

n number of electron in reduction of a molecule

D diffusion coefficient

C concentration

m rate of the mercury flow in capillary

t lifetime of a drop of mercury (2 to 7 sec.).

n,D,m and t are constants

($i_d = k\, C$)

m2/3 t1/6 is known by the capillary characteristics it depends on
1- the mercury column height above the capillary tube
2- the internal capillary dimensions.

A) **Advantages of DME**

1-The current voltage curve shows only the process

2- can be done in acidic solutions as Large overvoltage is needed for reduction of H+

3- reproducible results are obtained as Mercury electrode surface is continuously renewed, smooth surface of the mercury drop which allows reproducible rapid electron transfer. .

4- several runs can be performed using the same solution
as the surface area of the electrode is very small the
amount electrolyzed is negligible and the concentration
of the original solution nearly remains the same

5- The reduced metals at the electrode surface form
amalgum

Disadvantage of the DME:

1) potential above 0.4 V

Hg metal is oxidized with a production of a wave that interferes with the analyte.

2-The drop surface area is changeable

3-The drop surface area change by of potential change

Application of polarography

organic
Polarography

In organic
Polarography
-**Cations**
-**Anions**
-**Molecules**

1- Cations

a- No interference in $E_{1/2}$:

-Mixture of Cu^{+}, Cu^{2+}, Cd^{2+} Ni^{2+} Zn^{2+} Mn^{2+} is determined simultaneously in 0.5 M NH_4OH, O.5 M NH_4Cl as each cation has its characteristic $E_{1/2}$ and shows separate wave.

b- interference in $E_{1/2}$:

1-Pb^{2+}, Ti^{+} and Sn^{2+} the same $E_{1/2}$ (-0.5V) in neutral and acidic medium.

Use NaOH medium:

- **Pb^{2+} form a complex with $E_{1/2}$ -0.8 V**
- **Sn^{2+} can be oxidized to Sn^{4+} which is reduced at -0.35 V• Ti^{+} is reduced at -0.49.**

2- Cu^{2+} and Bi^{3+} both are reduced at -0.25 in HNO_3 .Use tartarate at pH 2 - 5 the potential is altered to -0.15 for Cu^{2+} and - 0.37 for Bi^{3+}

Amperometry

Amperometry

Definition

Amperometry refers to the measurement of current under a constant applied voltage and under
these conditions it is the concentration of analyte which determine the magnitude of current.

In Amperometric titration the potential applied between the indicator electrode (dropping mercury electrode) and the appropriate depolarizing reference electrode (saturated calomel electrode) is kept constant and current through the electrolytic cell is then measured on the addition of each increment of titrating solution.

In these titrations the current passing through the cell between the indicator electrode and
reference electrode at a suitable constant voltage is measured as a function of the volume of the titrating reagent.

By diffusion:
-occurs due to concentration gradient of ions
-The rate of mass transport by diffusion depends on the
concentration and the diffusion coefficient (a constant
value characteristic for the analyte)
-the transport (current) will depend on concentration.
=Small ion conc. small inflection of curve
=high ion conc. large inflection of curve
in polarographic analysis the mode of mass transport should be only by diffusion.

Ilkovič Equation:

Id = 607 n D1/2 C m2/3 t1/6

Id **average diffusion current**

n: number of electron in reduction of a molecule

D: diffusion coefficient

C: concentration

m rate of the mercury flow in capillary

t lifetime of a drop of mercury (2 to 7 sec.).

n,D,m and t are constants

(id = k C)

Principle:

According to Ilkovic equation

(Id= 607 X n X D1/2 X m2/3 X t 1/6 X C) ,

the diffusion current

(= limiting current - residual current) is directly proportional to the concentration of the electroactive

material in the solution.

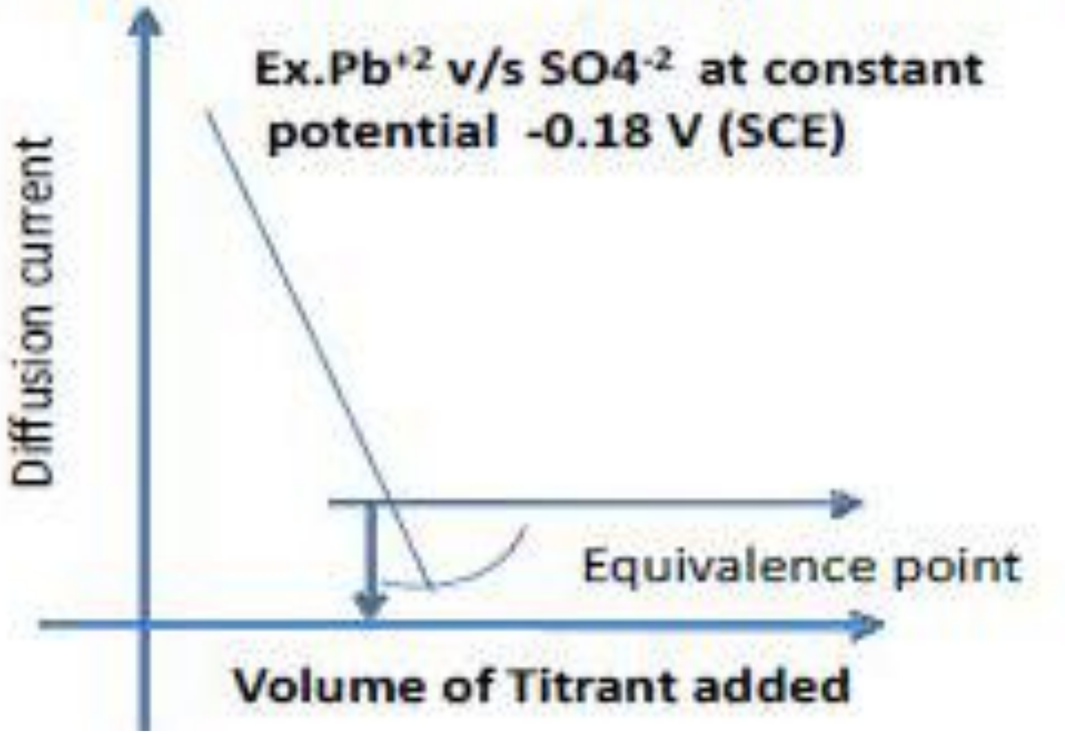
If some of the electro-active material is removed by interaction
with reagent, the diffusion current will decrease. This is the fundamental principle of
amperometric titrations. The observed diffusion current at a suitable applied voltage is measured
as a function of the volume of the titrating solution: the end point is the point of intersection of
two lines giving the change of current before and after the equivalence point

Titration Curves in Amperometry:

- **Titrand + Titrant --→-- Product.**

A) **Titrand is reducible but titrant and product not:** When solution containing Pb^{+2} ion is titrated against SO_4^{-2} ion. A precipitate of $PbSO_4$ is formed. The titration can be performed at fixed potential -0.8 Volt v/s saturated calomel electrode.

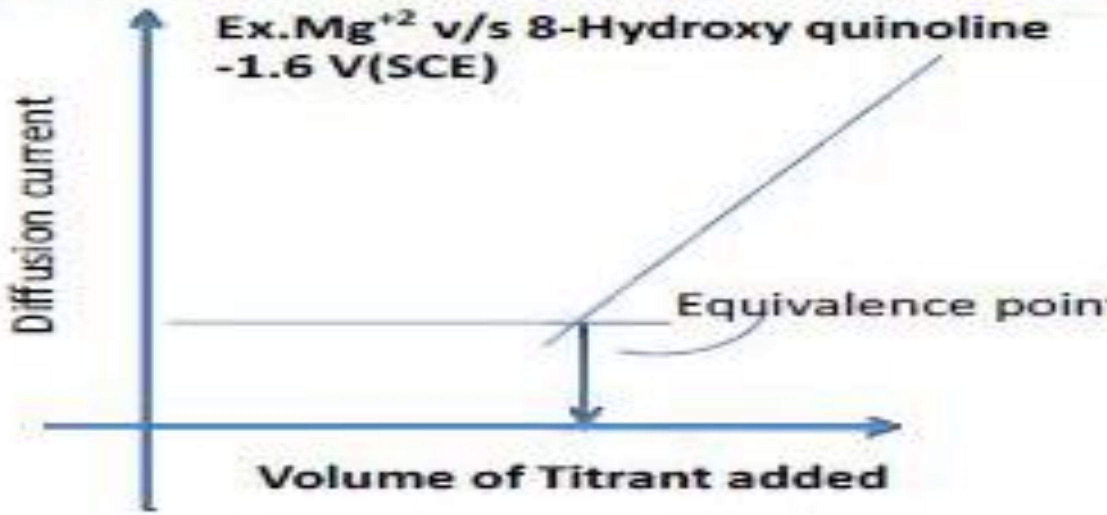
A) Ttrand is reducible but titrant and product not.



B) Titrant is reducible but titrand and product not : When solution containing Mg+2 ion is
titrated against with the reducible species such as 8- hydroxy quinoline because Mg+2 ion does
not undergoes reduction. Beyond the end point the 8- hydroxyl 4quinoline undergoes reduction.

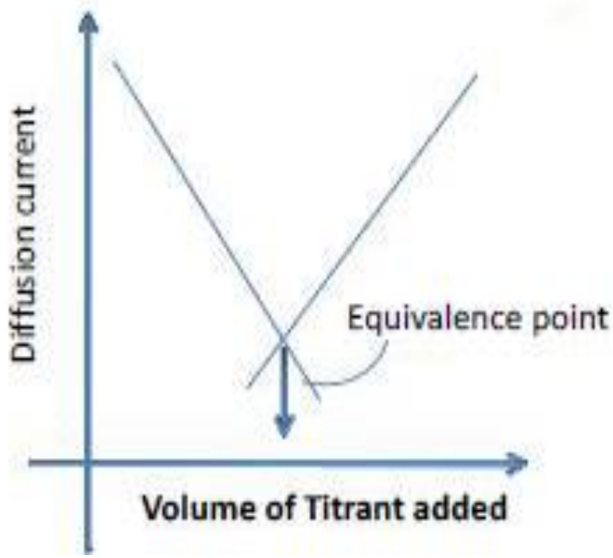
As its concentration increases diffusion current also increases.

B) Titrant is reducible but titrand and product not



C) Titrand and titrant both are reducible but product not : When solution containing Pb^{+2} ion is titrated against $K_2Cr_2O_7$. The titration is performed at potential of -0.8 Volt v/s SCE . Diffusion current is decreases due to removal of Pb^{+2} ion. The current is minimum at the end point. On further addition of the titrant the current once again increases. V shaped curve is obtained.

C) Ttrand and titrant both are reducible but product not Ex. Pb^{+2} v/s $K_2Cr_2O_7$ at constant potential -0.8 V (SCE)



Contents:

1.Conductometry-:
➢Introduction
➢Ohm's law.
➢Conductometric measurements.
➢Factor affecting conductivity.
➢Applications of conductometry.

2.Conductometric titration-:
➢ Introduction.
➢Types of conductometric tiration.
➢Advantages of conductometric tiration.
3.Recent devlopement
4.References.

INTODUCTION:

- It is an electrochemical method of analysis concerned with electrical conductance through an electrolyte solution .

(or)

- It is defined has as determination or measurement of the electrical conductance of an electrolyte solution by means of a conductometer .

- electric conductivity of an electrolyte solution depends on :

١. Type of ions (cations, anions)
٢. Concentration of ions
٣. Temperature
٤. Mobility of ions

Conductance equal 1/R

The units of conductance = moh or ohm-1

- Conductometry means measuring the conductivity of ionic solutions caused by mobility of ions towards respective electrodes in presence of an electric field.
- Conductivity is measured by using conductometer. Units of conductivity is mhos(Ω^{-1}).
- Conductivity is generally measured by using a Wheatstone bridge circuit and a conductivity cell made of platinum.

$$R = V/i$$

V-potential difference in volts

i-current in amperes

$$C = 1/R$$

Total conductance of the solution is directly proportional to the sum of the n individual ion contributions .

$$G = \sum c_i \Lambda_{m,i}$$

Ohm's law-

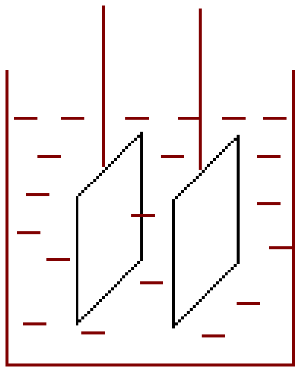
The magnitude of conductometric titration is based on ohm's law.

$$i = e/R$$

where

i = current in amperes
e = potential difference
R = resistance in ohm's

Conductivity measurements



1.Electrodes

Two parallel platinized Pt. foil electrodes or Pt. black with electrodeposited a porous Pt. film which increases the surface area of the electrodes and further reduces faradaic polarization.

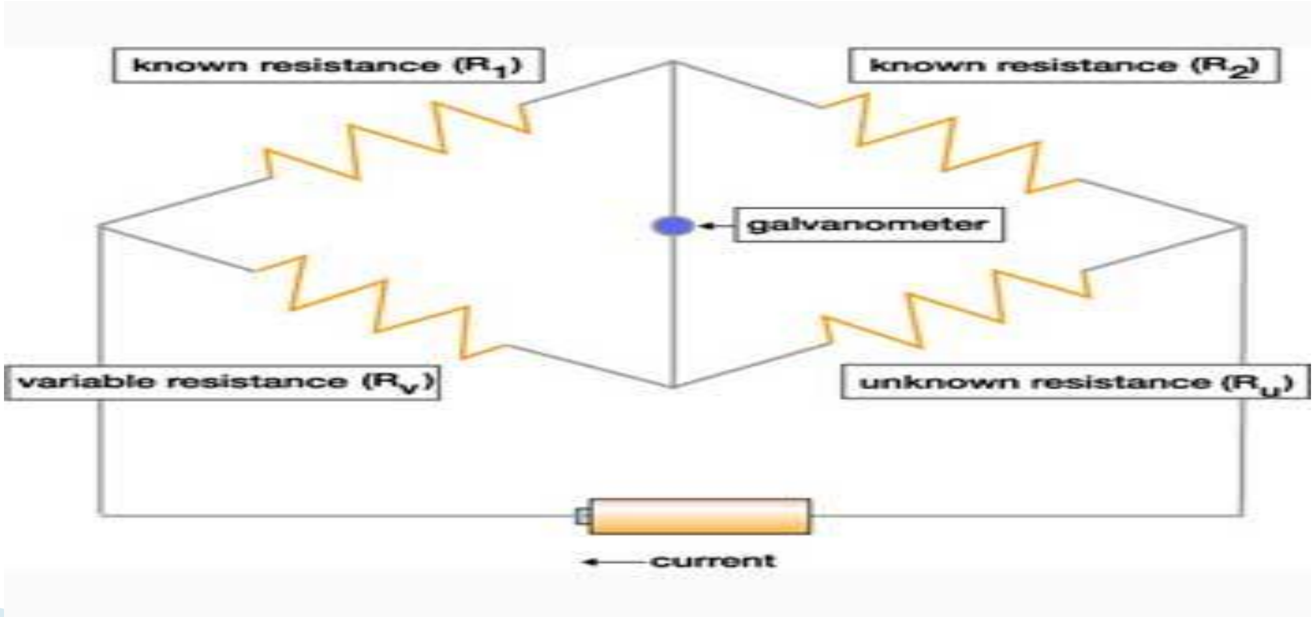
2.Primary standard solutions

Primary standard KCl solution ,at 25°C, 7.419g of KCl in 1000g of solution has a specific conductivity of 0.01286Ω^{-1}/cm.

3. **Conductivity Cell** :

4.Wheat stone bridge :

Avoid the change of temperature during determination



Factors affecting conductivity:

- Size of ions
- Temperature
- Number of ions
- Charge of ions

- ✓ Specific conductivity:-It is conductivity offered by a substance of 1cm length and 1sq.cm surface area. units are mhos/cm.
- ✓ Equivalent conductivity:-it is conductivity offered by a solution containing equivalent weight of solute in it.

Molar conductance of various ions at infinite dilution at 25°C

ions	molar conductance
K^+	73.52
Na^+	50.11
Li^+	38.69
H^+	349.82
Ag^+	61.92
Cl^-	76.34
Br^-	78.4
OH^-	198

APPLICATIONS OF CONDUCTOMETRY

It can be used for the determination of:-

- Solubility of sparingly soluble salts
- Ionic product of water
- Basicity of organic acids
- Salinity of sea water (oceanographic work)
- Chemical equilibrium in ionic reactions
- Purity of distilled and de - ionised water can determined
- Conductometric titration

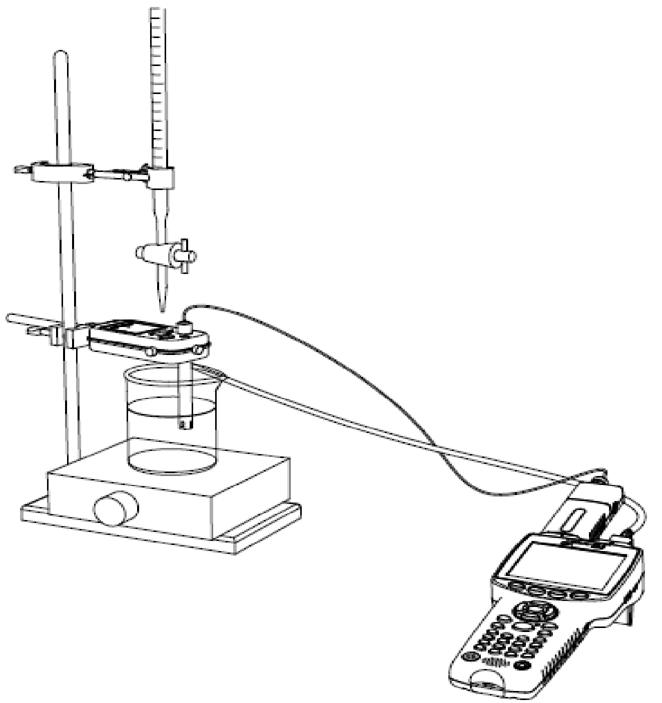
CONDUCTOMETRIC TITRATIONS:

:

Is a process of quantitative chemical analysis in which conc of a sample is determined. Which is done by adding a reagent(titrant) of known conc in measured volumes to the sample (anylate)

CONDUCTOMETRIC TITRATIONS:

- The determination of end point of a titration by means of conductivity measurements are known as conductometric titrations.



TYRES OF CONDUCTOMETRIC TITRATIONS:

- Acid –base or neutral titrations
- Replacement or displacement titrations
- Redox titrations
- Precipitation titrations
- Complexometric titrations
- Non-aqueous titrations

١. ACID- BASE OR NEUTRAL TITRATIONS:

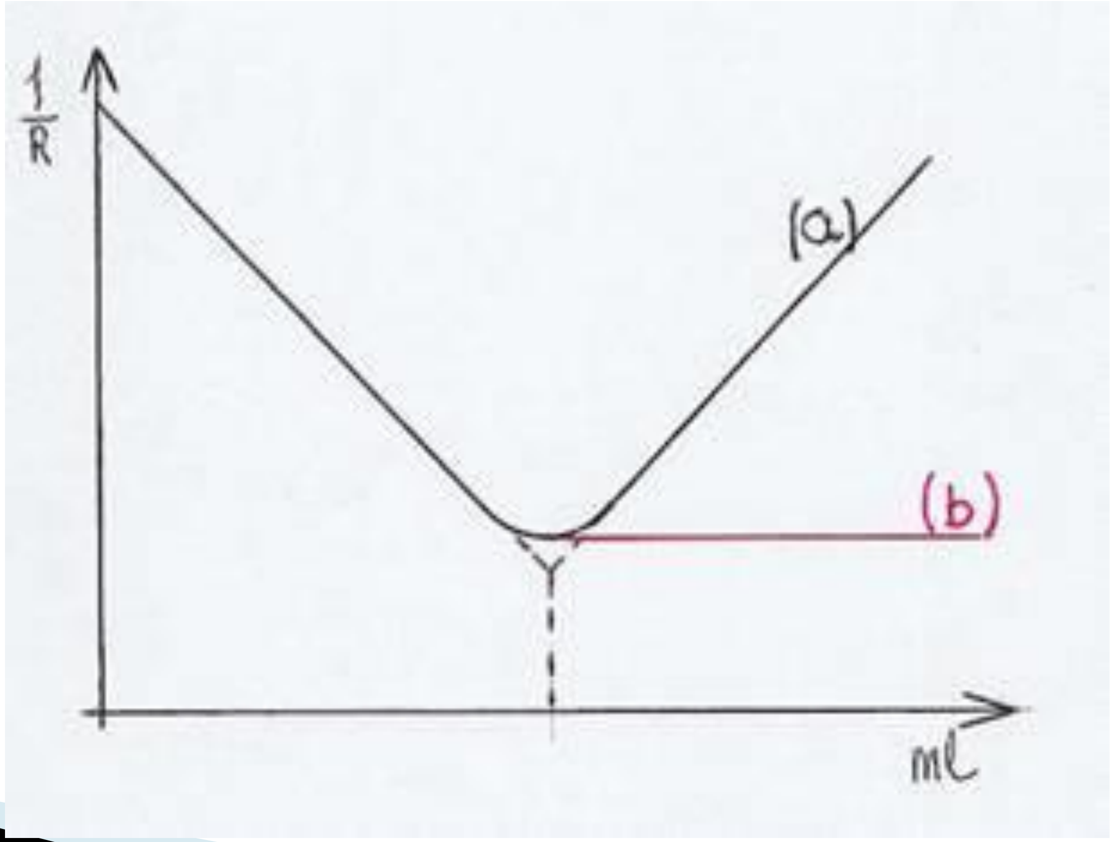
- STRONG ACID–STRONG BASE
  - e.g.:HCL vs NaOH
- STRONG ACID–WEAK BASE
  - e.g.:HCL vs NH4OH
- WEAK ACID–STRONG BASE
  - e.g.: CH3COOH vs NaOH
- WEAK ACID –WEAK BASE
  - e.g.: CH3COOH vs NH4OH

ACID-BASE TITRATIONS

- **Titration of strong acid**

(a) with **strong base e.g. HCl** with **NaOH**

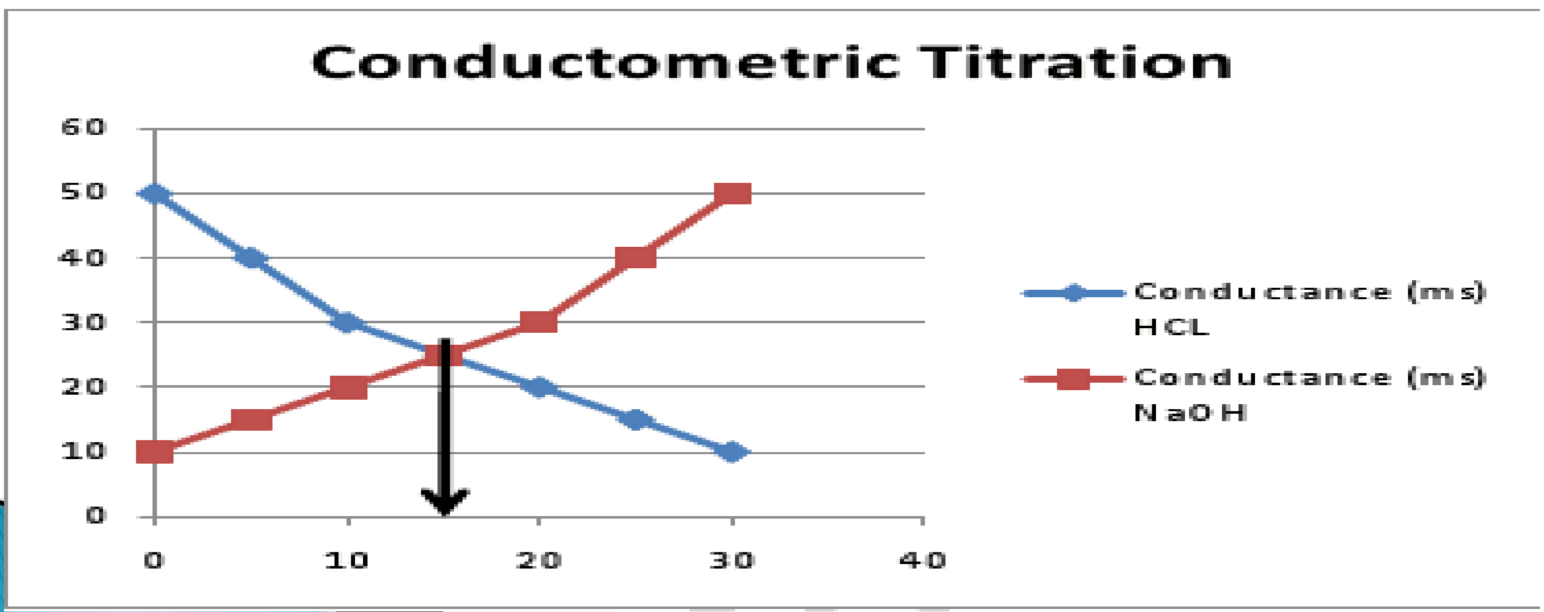
(b) with **weak base** **e.g. HCl** with **NH_4OH**



Strong acid vs strong base:

- Fall in conductance due to replacement of high conductivity Hydrogen ions by poor conductivity sodium ions
- Rise in conductance due to increase in hydroxyl ions

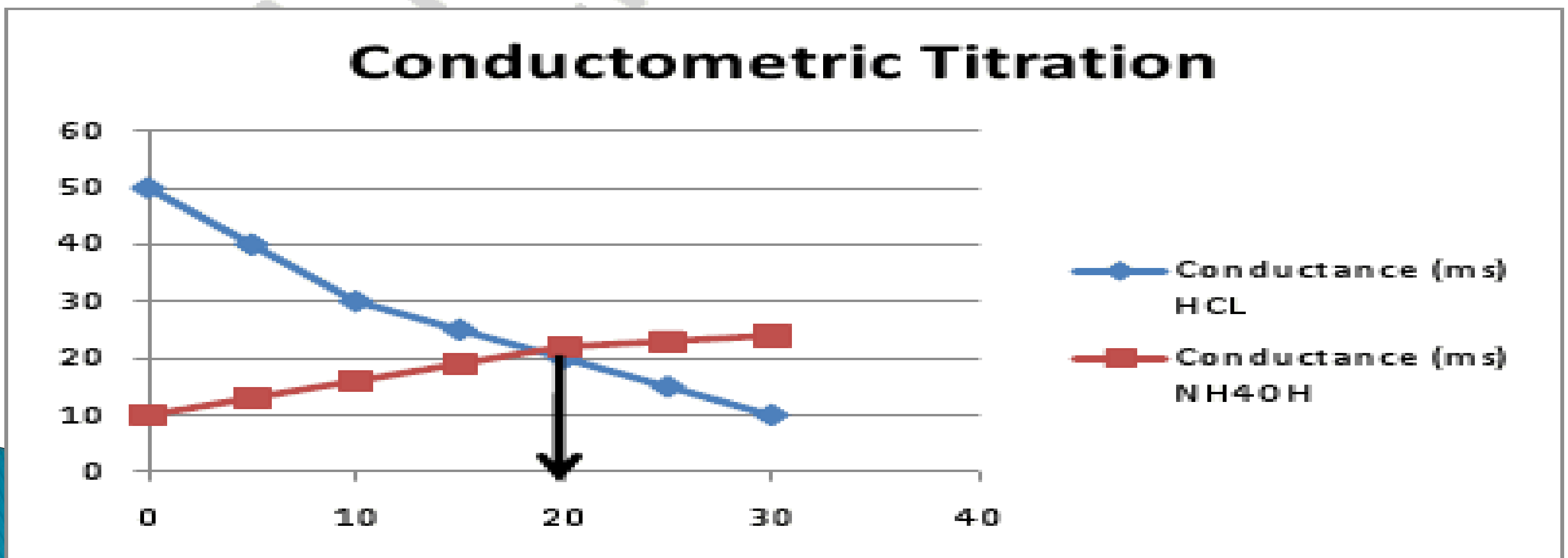
Strong Acid-Strong Base



Strong acid– weak base:

- Fall in conductance due to replacement of hydrogen by ammonium ions
- Conductance remain constant due to supression of NH4OH by NH4CL

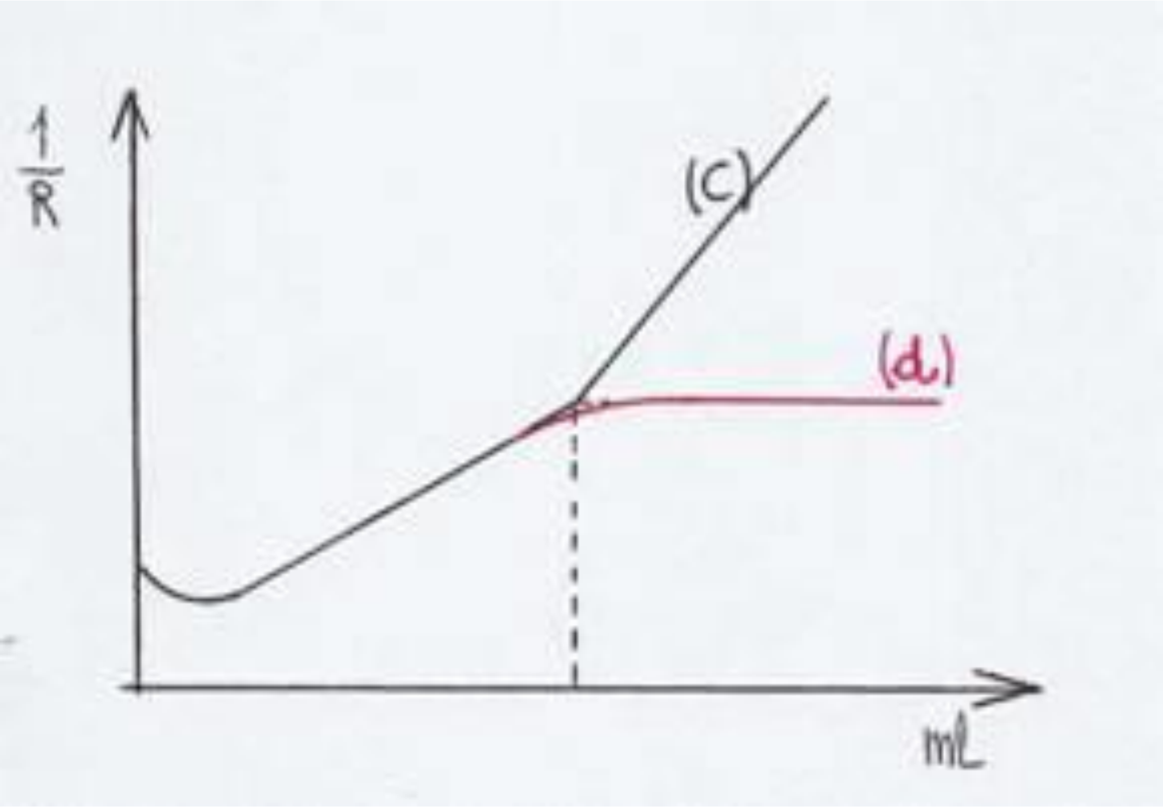
Strong Acid-Weak Base



- **Titration of weak acid**

(c) with **strong base e.g. CH_3COOH** with **NaOH**

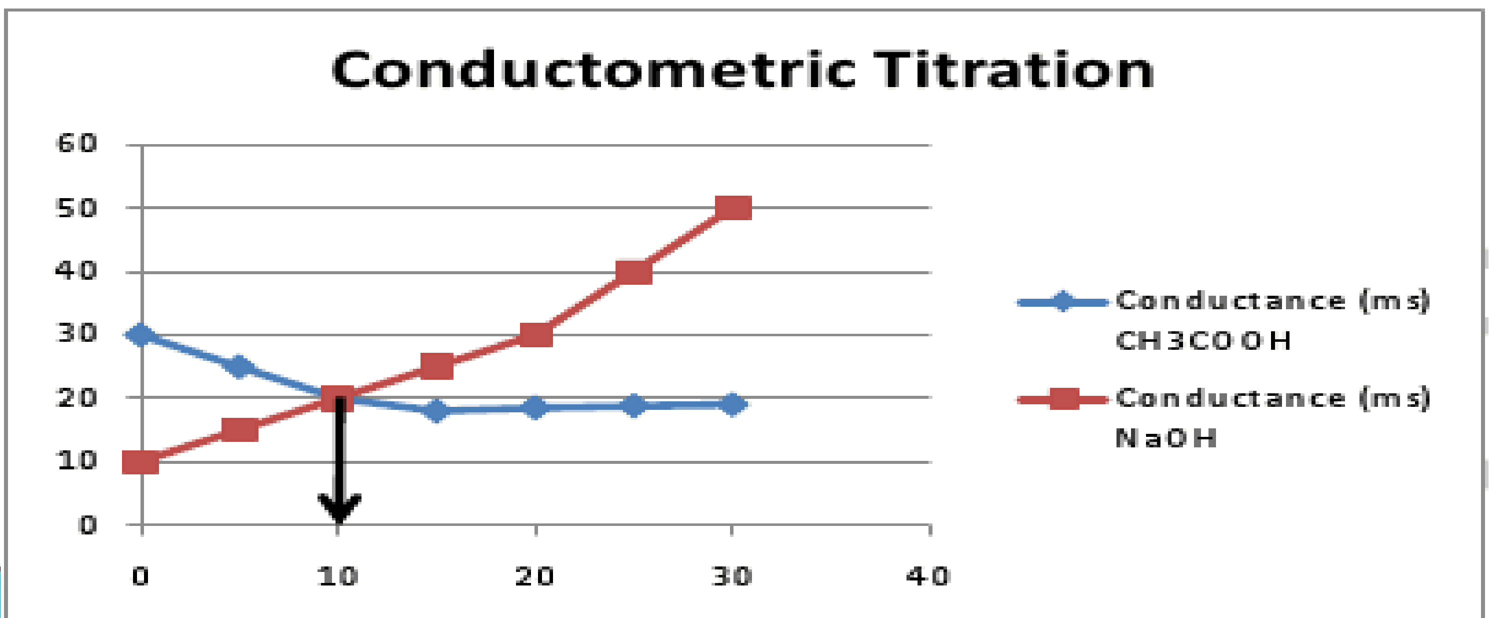
(d) with **weak base e.g. CH_3COOH** with **NH_4OH**



Weak acid –Strong base:

- Initial decrease in conductance followed by increase due to NaOH
- Steep rise due to excess of NaOH

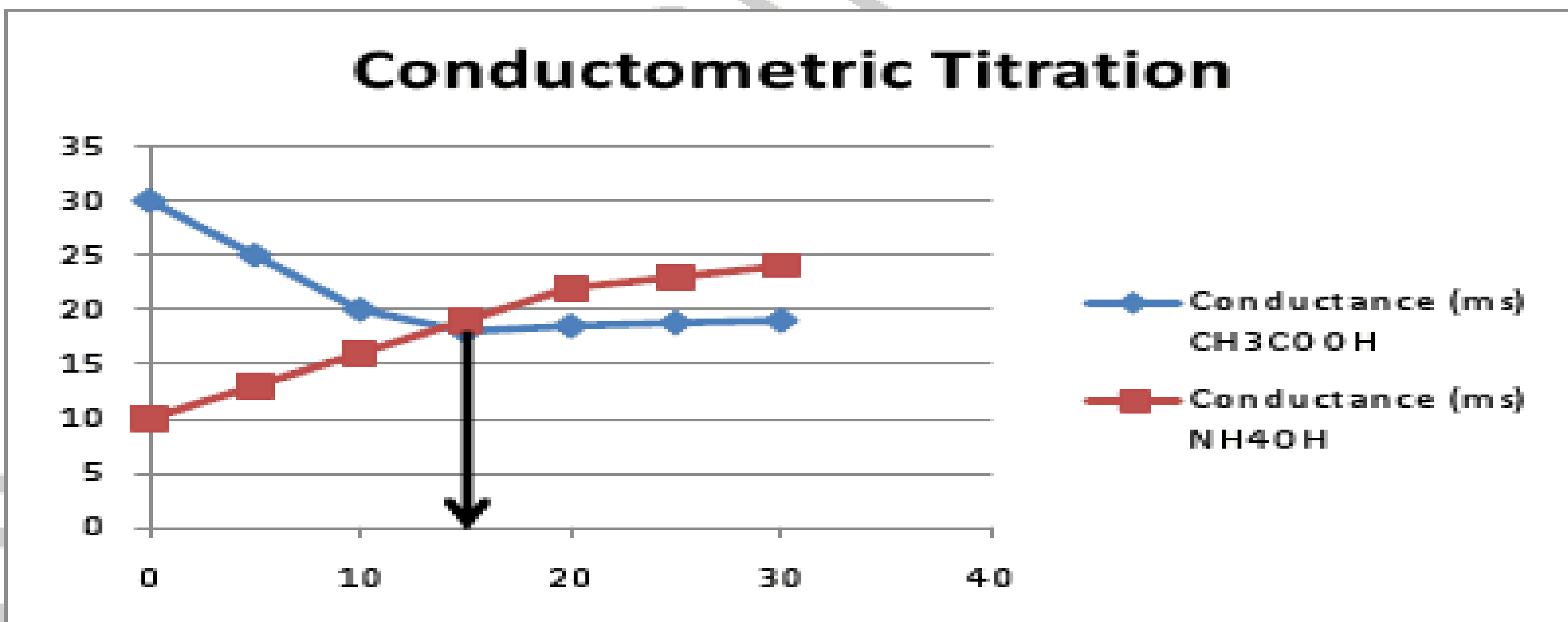
Weak Acid-Strong Base



Weak acid- weak base:

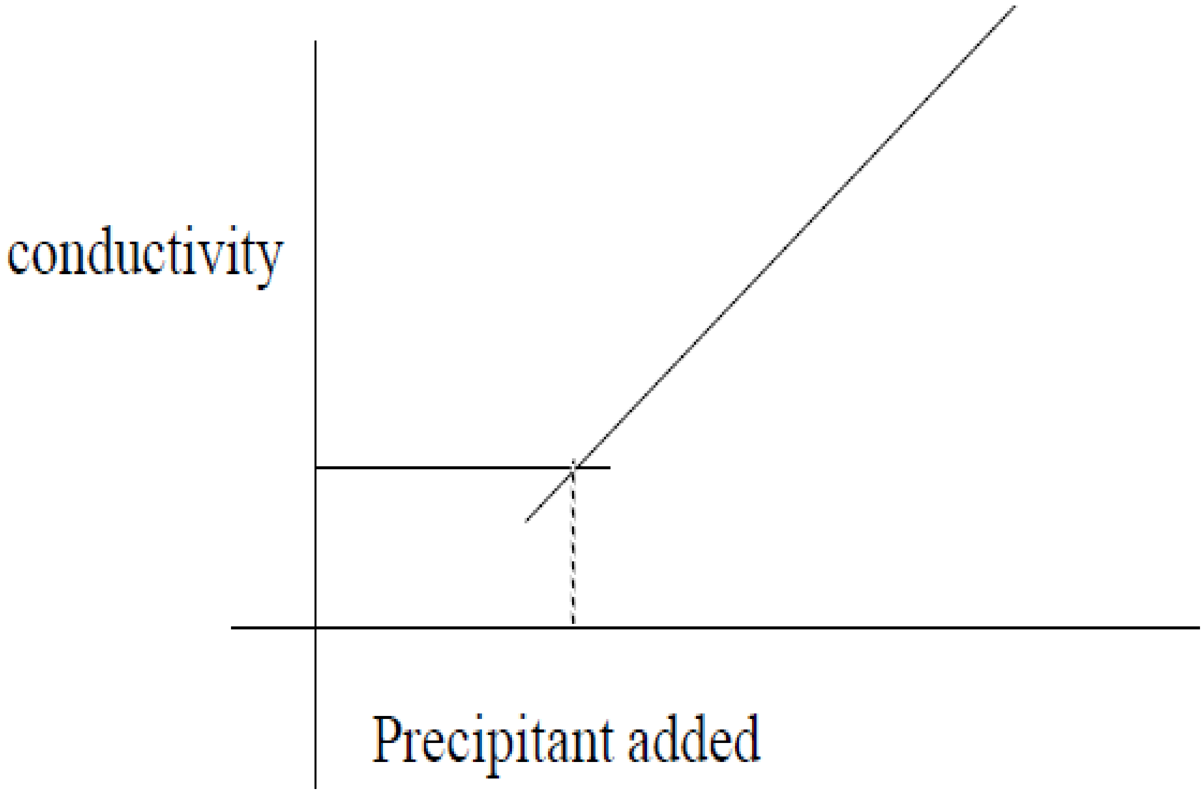
- Increase in conductance due to excess of CH3COOH
- Constant conductance due to supression of NH4OH by CH3COOH

Weak Acid-Weak Base



PRECIPITATION TITRATIONS:-

$[K^{+}+Cl^{-}]+[Ag^{+}+No_{3}^{-}]$



REPLACEMENT TITRATIONS

- Salt of strong acid and weak base vs. strong base

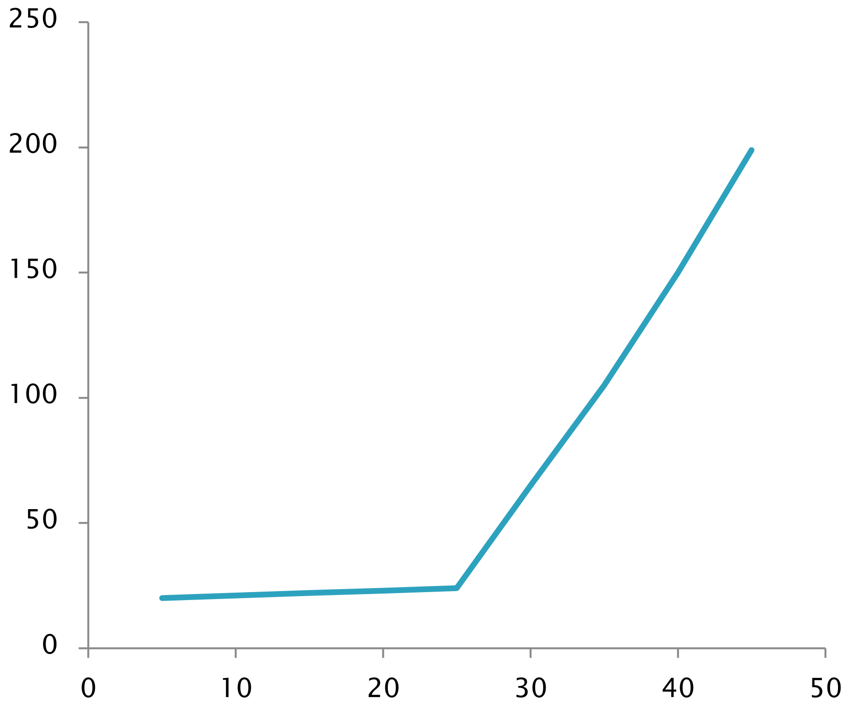
Ex: ammonium chloride vs. sodium hydroxide

- Salt of strong base and weak acid vs. strong acid

Eg: sodium acetate vs. hydrochloric acid

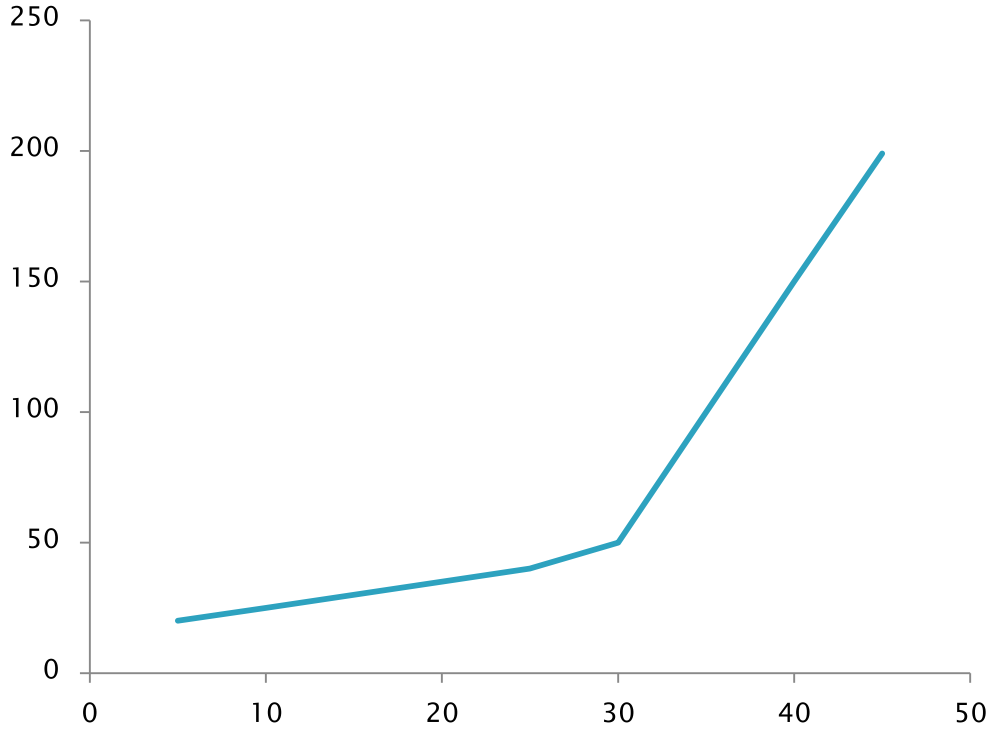
a)Salt of strong acid, weak base vs. strong base

$$NH_4Cl + NaOH \rightarrow NH_4OH + NaCl$$



b)Salt of strong base and weak acid vs. strong acid

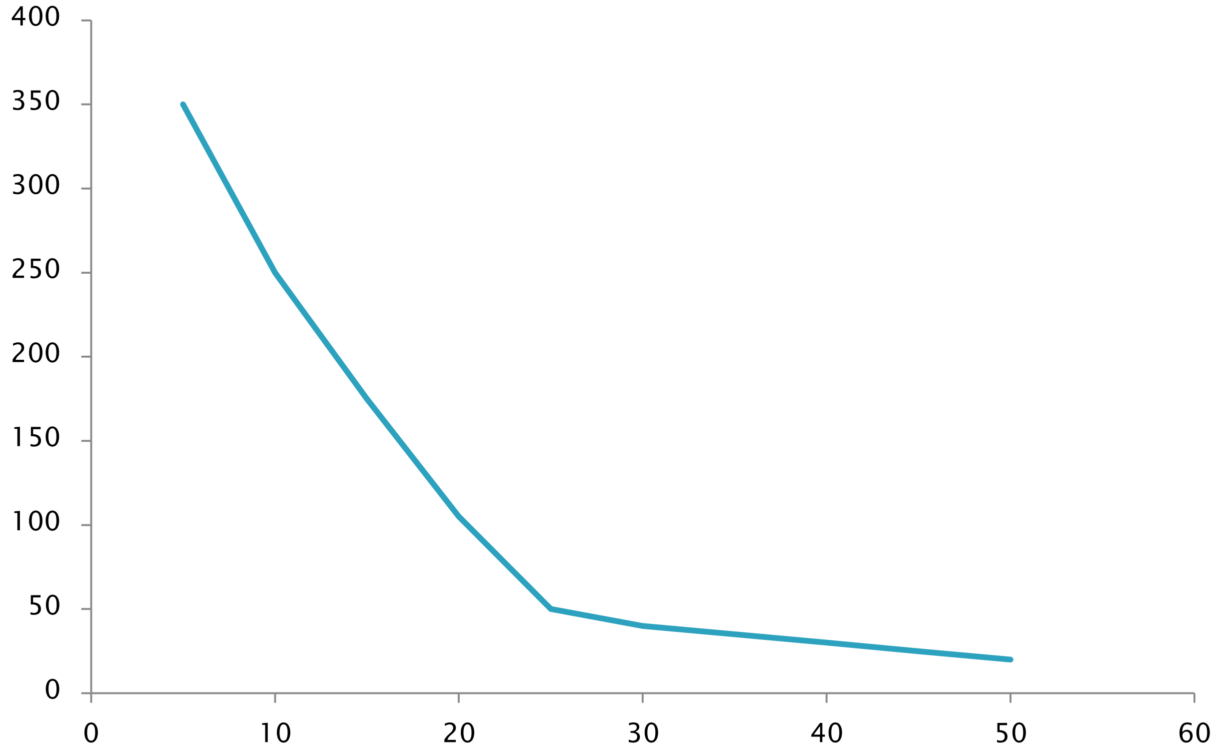
$$CH_3COONa + HCl \rightarrow CH_3COOH + NaCl$$



REDOX TITRATION

Titration of ferrous ions with dichromate ions:

$$6\,Fe^{2+} + Cr_2O_7^{2-} + 14H^+ \rightarrow 6Fe^{3+} + 2Cr^{3+} + 7H_2O$$



COMPLEXOMETRIC TITRATION

-KCl vs. $Hg(ClO_4)_2$

- Non-aqueous titrations can also be measured using conductometry.

a)titration of weak bases vs. perchloric acid in dioxan-formic acid.

b)Titration of weak organic acids in methanol vs. tetra methyl ammonium hydroxide in methanol-benzene.

ADVANTAGES OF CONDUCTOMETRIC TITRATIONS

- No need of indicator
- Colored or dilute solutions or turbid suspensions can be used for titrations.
- Temperature is maintained constant throughout the titration.
- End point can be determined accurately and errors are minimized as the end point is being determined graphically.

Disadvantages of conductometric titration:

١. **Increased level of salts in solution masks the conductivity changes , in such cases it does not give accurate results

٢. **Application of conductometric titrations to redox systems is limited because, high concentrations of hydronium ions in the solution tends to mask the changes in conductance

SOLVENT EXTRACTION

THE LECTURE IS CLASSIFIED TO:

1) Introduction

2) Basic Principles of Solvent extraction Method

3) The important of Solvent Extraction

4) Classification of Extraction **Systems**

5) methods of Extraction

6) Factors Influencing the Extraction Efficiency

7) Analytical Applications

1-1) **Introduction To Solvent Extraction**

Solvent extraction is a technique extensively utilized in both industrial applications and in the laboratory. It includes a variety of techniques such as liquid-liquid extraction (LLE), Liquid-solid extraction (LSE), supercritical fluid extraction (SFE), and other special techniques.

LLE is an extraction technique applied to liquids,-liquid samples, or samples in solution, using a liquid extracting medium.

1-2) ***Introduction To Solvent Extraction***

The quality of manufactured products often depends on proper chemical proportions, and measurement of the constituents is a necessary part of quality control [1].

Solvent extraction technique is a part of analytical chemistry and has been

recognized as an excellent separation method because of its ease, simplicity,

speed, and wide scope.

---

1) G. D. Christian, Analytical chemistry 6th Ed. (2004).

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2-1*) BASIC PRINCIPLES OF SOLVENT Extraction Method*

An extractant, is a substance primarily responsible for the transfer of a solute (here metal) from one phase to the other.

The extractant is dissolved in a suitable diluent and together act as a solvent. The diluent is immiscible with other phase which is usually water.

2-2) BASIC PRINCIPLES OF SOLVENT EXTRACTION METHOD

The extractant reacts with the solute by solvation/chelation/ion pair formation etc. to extract from the aqueous phase.

The distribution equilibrium between two phases is governed by Gibbs phase rule, given by:

GIBBS PHASE RULE,

P+V=C+2 (1)

Where,

P = is the number of phases,

V = is the variance or degree of freedom and

C = is the number of components

And the number *2* corresponds Temp. and Pressure .

GIBBS PHASE RULE,

In solvent extraction, we have

P = 2

two phases namely aqueous and organic phase,

the component C=1, viz. solute, in solvent and water phase and at constant temperature and pressure P=1, thus, we therefore have

2+1=1+2 i.e. P+V= C+2 ……….(2)

,

Nernst Distribution Law

According to Nernst distribution law,

If $[X]_1$ is concentration of solute in phase 1 and $[X]_2$ is the concentration of solute in phase 2 at equilibrium:

$K_D = [x]_1 / [x]_2$ ………….. (3)

Where K_D is called as the partition coefficient or distribution coefficient

The Partition Coefficient Or Distribution Coefficient

this partition or distribution coefficient **(K_D)**

is independent of the total solute

concentration in either of the phases

Distribution Ratio (D)

The distribution of a solute between two immiscible solvents in contact to eac other can be described by the distribution Ratio (D)

D = [X]1 / [X]2

Where [X] represents the stoichiometric or formal concentration of a substance X

and the subscripts 1 and 2 refer to the two phases.

Distribution Ratio (D)

Since in most cases, two-phase

system is of analytical interest, an organic solvent and aqueous are involved, D will be understood to be; $D = [X]_{org} / [X]_{aq}$

The subscript org. and aq. refer to the organic and aqueous phases **respectively**

Distribution ratio ‘D’ is dimensionless quantity, separation of two solutes by solvent extraction is expressed by the term, separation factor (α),

which is related to individual distribution ratios,

Separation Factor

$\boldsymbol{\alpha} = D_A / D_B$

D_A and D_B are

the respective distribution ratios of

solute A and B.

Percent Extraction (%E)

The more commonly used term for expressing the extraction efficiency by analytical chemist is the percent extraction "E", which is related to "D" as

$$\%\,Extraction\;(E) = \frac{(100D)}{D + Vaq/Vorg}$$

Where, V represent solvent volume and the other quantities remain as previously defined.

The percent extraction may be seen to vary with the volume ratio of the two phases as well as with D.

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3) ***Solvent Extraction May Serve***
the following three purposes:

i) Preconcentration of trace elements

ii) Elimination of matrix interference

iii) Differentiation of chemical species.

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4) Classification Of Extraction Systems

The classification of extraction systems is based upon the process of extraction.

Thus, based upon the process of extraction,

extraction systems can be classified into

four major classes

Classification Of Extraction Systems

a) Chelate extraction

b) Extraction by solvation

c) Extraction involving ion pair formation

d) Synergic extraction

(a -1)
Chelate Extraction

In this class, extraction proceeds by the process of formation of chelate or

closed ring structure between the chelating agent and the metal ion to be extracted. e.g.

i) The extraction of Uranium with

8-hydroxyquinoline in chloroform.

.

(a-2) Chelate Extraction

ii) The extraction of Iron with cupferron in carbon tetrachloride

the ammonium salt of *N*-nitroso-N-phenylhydroxylamine, is a common reagent for the complexation of metal ions. Its formula is $NH_4[C_6H_5N(O)NO]$. The anion binds to metal cations through the two oxygen atoms, forming five-membered chelate rings.

Cupferron is prepared from phenylhydroxylamine and an NO+ source:

b) Extraction By Solvation

<u>In this class,</u> the extraction proceeds by the process of solvation of the species which is extracted into organic phase. Oxygenated organic solvents such

as alcohols (C-OH), ketones, ethers and esters show some basicity because of the

lone pair of electron on the oxygen atom and can therefore directly solvate

protons and metal ions and bring about their extraction.

e.g. i) The extraction of Uranium with tributyl phosphate from nitric acid

ii) The extraction of Iron(III) with diethyl ether from hydrochloric acid.

C) Extraction Involving Ion Pair Formation

The extraction proceeds with the formation of neutral uncharged species which in turn gets extracted in to the organic phase. The best example of this is the extraction of Scandium and Uranium with trioctyl amine

from mineral acids.

In this case an ion pair is formed between complex of metal ion with high

molecular weight amine and anionic species of mineral acids.

D) Synergic Extraction

In this case, there is enhancement in the extraction on account of use of two extractants.

e.g. the extraction of Uranium with tributylphosphate (TBP) as well as 2-thionyltrifluroacetone (TTA).

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5) Methods Of Extraction

Three basic methods of liquid-liquid extraction are generally utilized in the analytical laboratory.

A) Batch Extraction

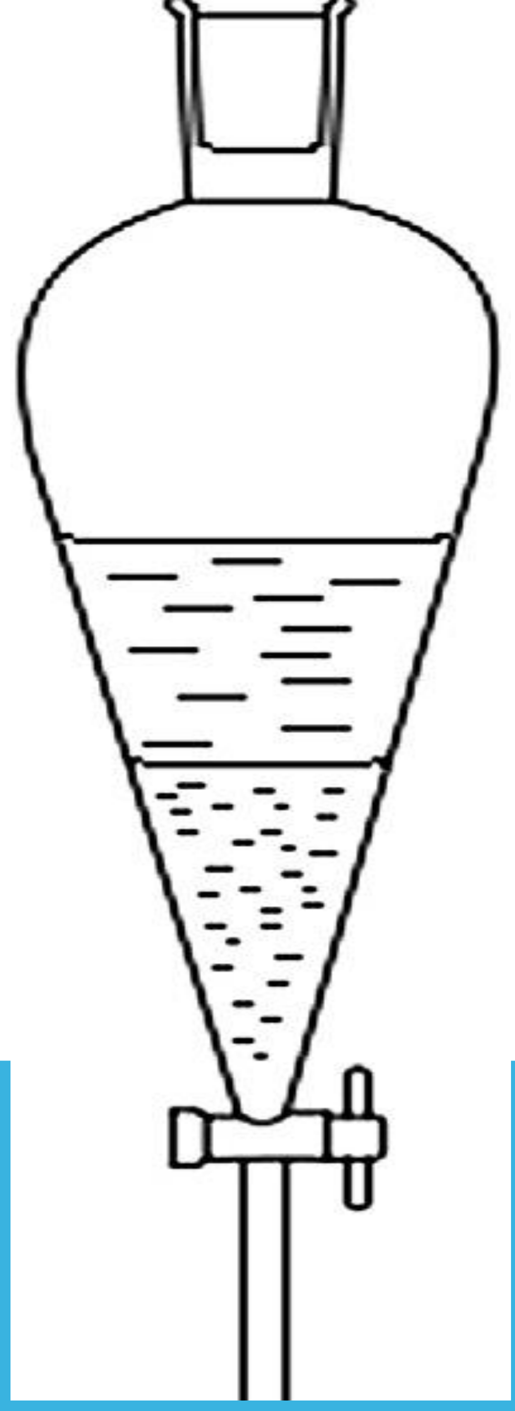
Batch extraction, the simplest and most commonly used method, consists of extracting the solute from one immiscible layer into other by shaking the two layers until equilibrium is attained, after which the layers are allowed to settle before sampling.

This is commonly used on the small scale in chemical laboratories**.**

The most commonly employed apparatus for performing a batch extraction is a *separatory funnel*.

The batch extractions may also be used with

advantage when the distribution ratio is large.



B-1) Continuous Extraction

The second type, continuous extraction, makes use of a continuous flow of

immiscible solvent through the solution or a continuous countercurrent flow of both phases.

Continuous extractions are particularly applicable when the

distribution ratio is relatively small.

Continuous extraction device operate on the

same general principle, which consist of distilling the extracting solvent from a boiler flask and condensing it and passing it continuously through the solution

being extracted.

B-2) Continuous Extraction

.**The extracting liquid separates out and flows back into the receiving flask, where it is again evaporated and recycled while the extracted solute remains in the receiving flask.**

When the solvent cannot easily be distilled, a continuous supply of fresh solvent may be added from a reservoir

C) Countercurrent Extractions

Extraction by continuous countercurrent distribution is the third general type and is used primarily for fractionation purposes.

The separation through continuous countercurrent method is achieved by virtue of the density difference between the fluids in contact. In vertical columns, the denser phase enters at the top and flows downwards while the less dense phase enters from the bottom and flows upwards. The choice of method to be employed will depend primarily upon the value of the distribution ratio of the solute of interest, as well as on the separation factors of the interfering materials.

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6) Factors Influencing The Extraction Efficiency

Primary requirement of solvent extraction for separation /removal purposes is

a high distribution ratio of the solute of interest between the two liquid phases.

It is useful to employ a number of different techniques for enhancing the distribution ratio.

It depends on the nature of the species being extracted and extraction system.

The attainment of selectivity in an extraction procedure is also very important.

Some of the factors, which affect the distribution of solute of interest, are given below.

6-1) Nonchemical Factors Affecting Extraction

factors address the nonchemical elements of the extraction, which include

(1) the choice of the extraction technique,

(2) the choice of solvent and aqueous phase volumes,

(3) the time of extraction,

(4) the solvent evaporation procedure, etc.

These factors are important for achieving the appropriate extraction efficiency for a successful utilization of LLE in sample preparation. The choice of the extraction procedure (batch or continuous), the number of extractions when using the batch procedure, etc.,

6-2) Factors Affecting Solvent Extraction

A) Choice of solvent

B) Acidity of an aqueous phase

C) Stripping

D) Use of masking agents

E) Salting-out agents

F-) Variation of oxidation state

G) Synergic Extraction

H) Use of organic acid media

Solvent Extraction (part 2)

efficiency

The lecture is classified to:

- **1) Introduction**
- **2) Basic Principles of Solvent extraction Method**
- **3) The important of Solvent Extraction**
- 4) Classification of Extraction Systems
- **5) methods of Extraction**
- **6)** Factors Influencing the Extraction Efficiency
- 7) Analytical Applications

Factors Influencing the Extraction Efficiency

- **Primary requirement of solvent extraction for** separation /removal purposes is a high distribution ratio of the solute of interest between the two liquid phases.
- It is useful to employ a number of different techniques for enhancing the distribution ratio.
- It depends on the nature of the species being
- extracted and extraction system.
- The attainment of selectivity in an extraction
- procedure is also very important.
- Some of the factors, which affect the
- distribution of solute of interest, are given below.

A) Choice of solvent

- safety, the toxicity and the flammability of the solvent must be considered.
- Use of a suitable solvent for effective separation is very important. Metal
- chelates and many organic molecules, being essentially covalent compounds do
- not impose many restrictions on the solvent and the general rules of solubility
- are the great use. In ion association systems and particularly in oxonium type
- ions, the role of solvents is very important. This is due to involvement of solvent
- in the formation of extractable species.

B) Acidity of an aqueous phase

- The extractability of metal complexes is greatly influenced by the acidity
- of an aqueous phase, so it is necessary to assure optimum concentration of H^+
- ions for maximum extraction. In the case of chelate extraction, the chelating
- reagent concentration is maintained constant; the distribution of the metal in a
- system is a function of pH. For this reason, curves of extractability versus pH at
- constant reagent concentration are of great analytical significance. Sometimes it
- is possible to achieve the desired characteristics of a solvent by employing a
- mixed solvent system.

C) Stripping

- Stripping is the removal of the extracted solute from the organic phase for
- further processing or analysis. In many colorimetric procedures and even
- radioactive techniques, the concentration of solute is determined directly in the
- organic phase. However, where further separation steps are required, it is
- necessary to remove the solute from the organic layer to more stable medium.

D) Use of masking agents

- In the extraction procedures for metal pairs that are difficult to separate;
- masking or sequestering agents are introduced to improve the separation factor.

E) Salting-out agents

- The term salting-out agent is applied to those electrolytes whose addition
- greatly enhances the extractability of complexes. The function of salting-out
- agent would be primarily of providing a higher concentration of complex and
- thus improve the extraction. Water is probably bound as a shell of oriented water
- dipoles around the ion and thus becoming unavailable as “free solvent”.
- Addition of salting-out agents decreases the dielectric constant

- Addition of salting-out agents decreases the dielectric constant of the
- aqueous phase, which favors the formation of the ion association complexes.
- Salting-out agents have been used with great success in separation involving the
- halide and thiocynate systems.

F) Backwashing

- Backwashing is an auxiliary technique used with batch extractions to
- influence quantitative separations of This technique is analogous in many respects to the re-precipitation step
- in a gravimetric precipitation procedure. With the proper conditions, most of the
- impurities can be removed by this backwashing operation, with neglisible loss of
- the main component, thereby attaining a selective operation.elements.

F-) Variation of oxidation state

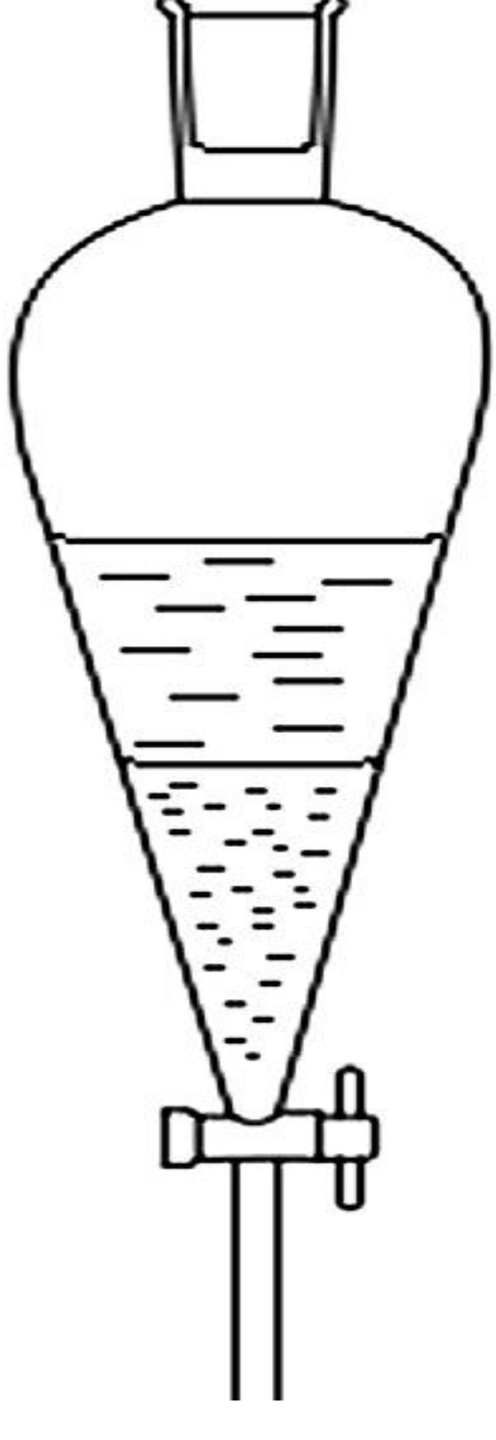
- The selectivity of an extraction is increased by the modification of
- oxidation states of the interfering ions present in solution, in order to prevent the
- formation of their extractable metal complexes e.g. reduction of Cerium(IV) to
- Cerium(III) prevents extraction of this element from nitrate media, the extraction
- of Iron(III) from chloride solutions can be prevented by reduction to Iron(II),
- which is not extractable. Similarly, Antimony(V) may be reduced to the
- tetravalent state to suppress its extraction.

G) Synergic Extraction

- Synergism is defined as the combined action of two complexing reagents,
- which is greater than the sum of the actions of the individual reagents used
- alone. An example of the synergic extraction of Ce(III) with picrolonic acid and benzo-15-crown-5.

H) Use of organic acid media

- Organic acid media are having ability of controlling the concentration of
- the complexing ligand, is one of the unique application, the ease of adjustment of
- pH and the wide difference in pH at which various metal ions form
- anionic complexes.



7) Applications

Removal of high boiling organics from wastewater
such aniline, phenols, nitrate aromatics have

Removal of carboxylic acid

Essential oil extraction

Agricultural chemical extraction

Agricultural chemicals such as herbicides and pesticides

Food industry applications

Removal of high boiling organics from wastewater

New technologies are developing day by day to reuse the water efficiently. Presence of micro pollutant such aniline, phenols, nitrate aromatics have adverse effect which renders the reuse of water. Solvent extraction method was reported as most effective method to remove and recover these chemicals from the wastewater.

Several extractants including octanol, amines, cyanex, diethyl carbonate, ionic liquid etc. has been employed to remove high boiling organics especially phenol from wastewater. [5].

Removal of carboxylic acid

Acetic acid is produced during fermentation of yeast which is an important inhibiting agents [11]. This acetic acid as well as other carboxylic acids and dicarboxylic acids
such as formic acid, succinic acid, valeric acid etc. are removed from aqueous stream using LLE process. LLE process is more economical and less energy consuming process compared to the distillation process [6].

Essential oil extraction

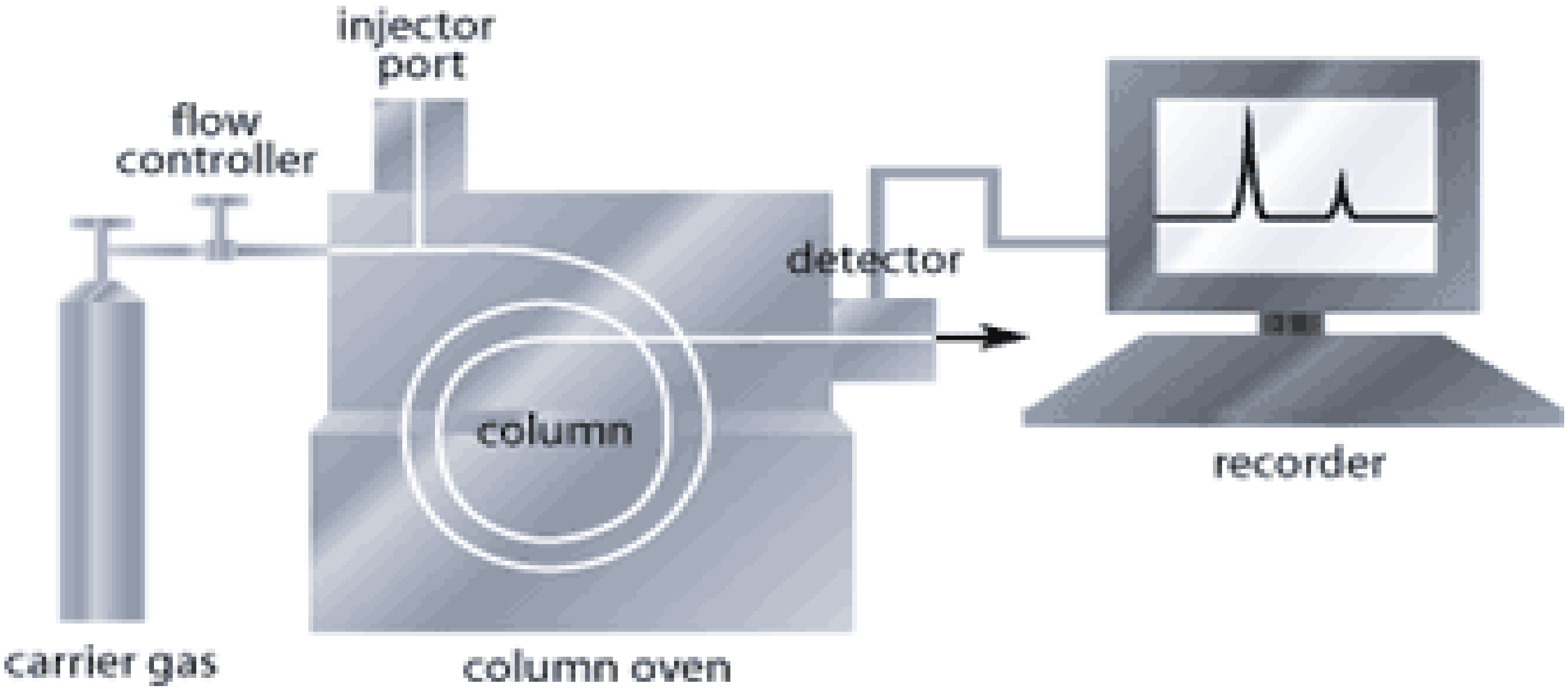
Bio-oil is produced from biomass pyrolysis. The end product is a complex mixture of different organic compounds. Due to high water content and high viscous property of bio-oil, LLE method is an efficient process to separate bio-oil according to their polarity and different chemical groups compared to the solid-phase extraction. The effect of extraction solvent and volume ratio is significant in case of LLE of bio-oil [8].

Agricultural chemical extraction
Agricultural chemicals such as herbicides and pesticides are extracted from the water using LLE method. Metals and mixture of organic compounds remains in the agricultural waste are separated through the solvent extraction process [9].

Food industry applications

LLE process is commonly used in food industries. As for example, separation and purification of a particular flavor or fragrance as well as caffeine extraction are done by this process [12].

Gas Chromatography



What is Gas Chromatography?

- It is also known as…

Gas-Liquid Chromatography (GLC)

GAS CHROMATOGRAPHY

- Separation of gaseous & volatile substances
- Simple & efficient in regard to separation

GC consists of:

GSC (gas solid chromatography)

GLC (gas liquid chromatography

GSC principle is **ADSORPTION**

GLC principle is PARTITION

Sample to be separated is converted into vapour

And mixed with gaseous M.P

Component more soluble in the S.P → travels slower

Component less soluble in the S.P → travels faster

Components are separated according to their **Partition Co-efficient**

Criteria for compounds to be analyzed by G.C

1.VOLATILITY:

2.THERMOSTABILITY:

<u>How a Gas Chromatography Machine</u>
⸮Works⸮

– **<u>First</u>**, a vaporized sample is injected onto the *chromatographic column*.

– **<u>Second</u>**, the sample moves through the column through the flow of inert gas.

– **<u>Third</u>**, the components are recorded as a sequence of peaks as they leave the column.

Chromatographic Separation

– Deals with both the *stationary*
the *mobile phase*. *phase* and

- **Mobile** – inert gas used as carrier.
- **Stationary** – liquid coated on a solid
within a column. or a solid

Chromatographic Separation

Chromatographic Separation
– **In the mobile phase**, components of the sample are uniquely drawn to the stationary phase and thus, enter this phase at different times
–
The parts of the sample are separated within the column.
Compounds used at the stationary phase reach the detector at unique times and produce a series of peaks along a time sequence.

The peaks can then be read and analyzed by a forensic scientist to determine the exact components of the mixture.

– Retention time is determined by each component reaching the detector at a characteristic time.

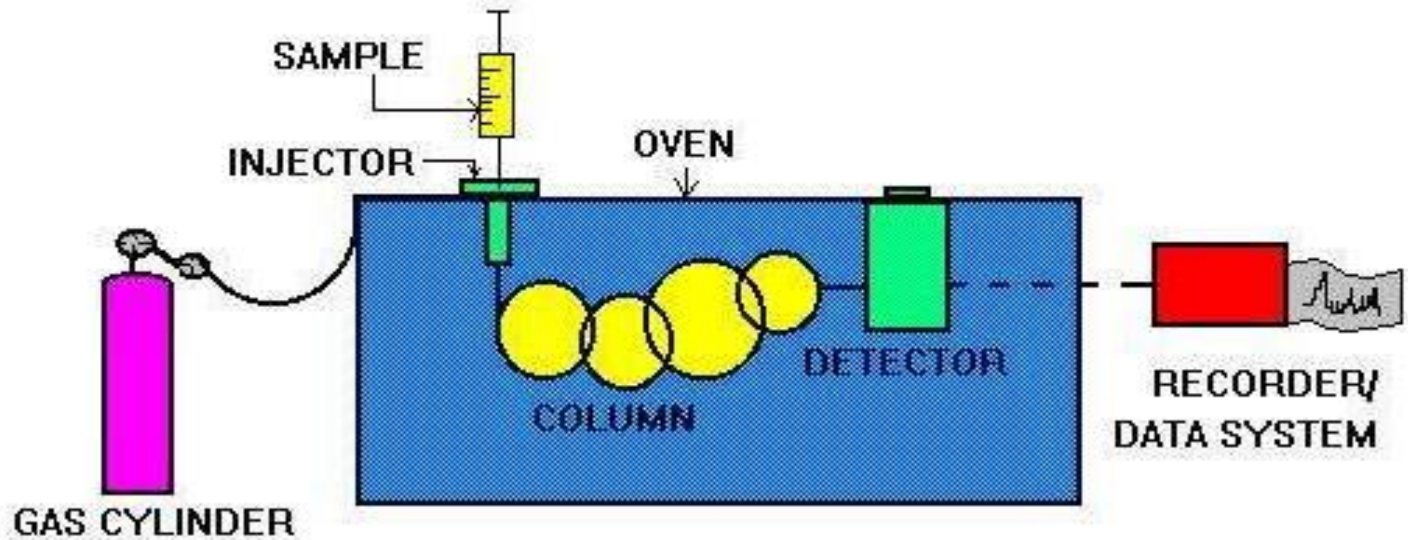
Chromatographic Analysis

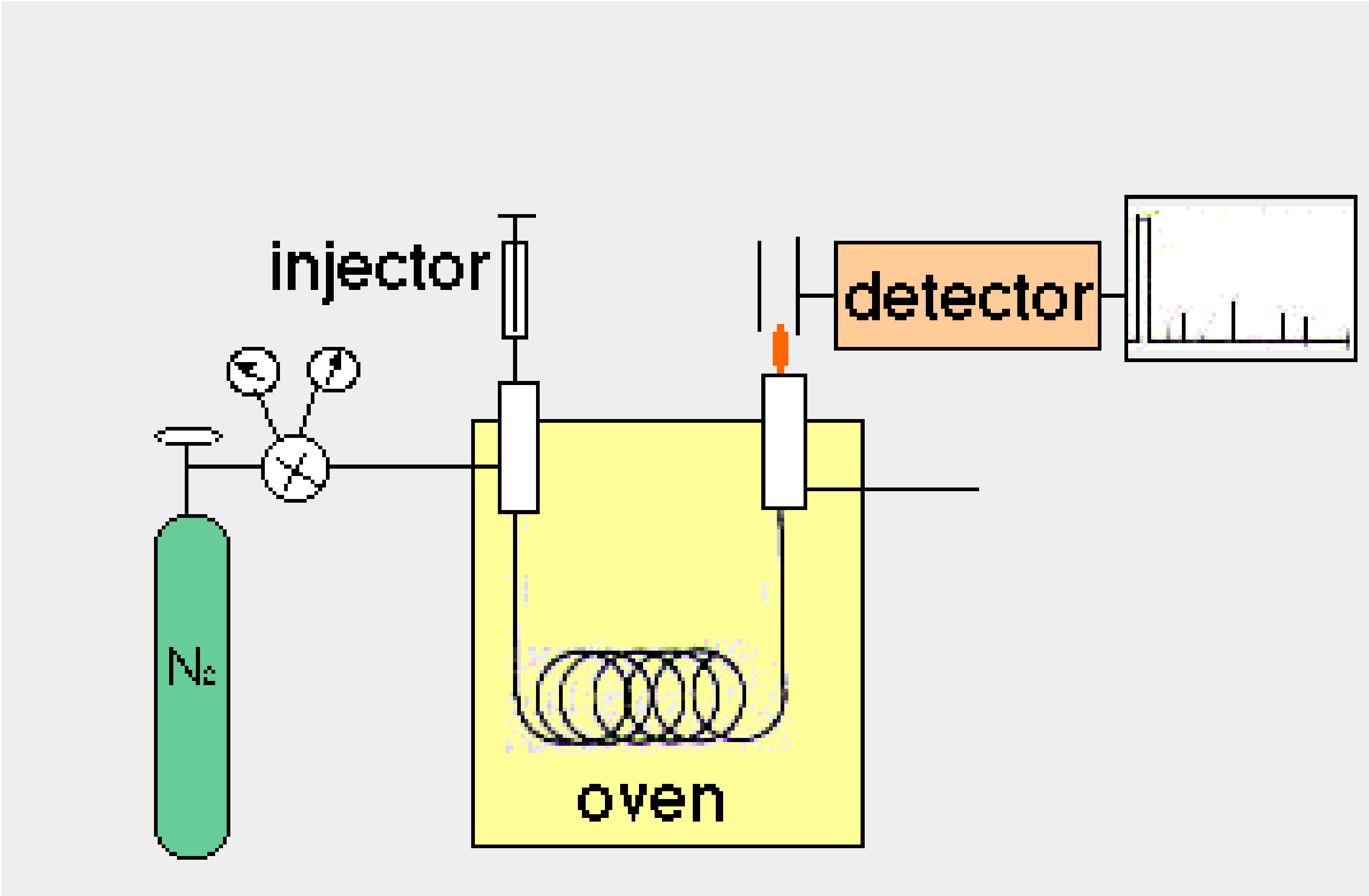
The number of components in a sample is determined by the number of peaks.

– The amount of a given component in a sample is determined by the area under the peaks.

– The identity of components can be determined by the given retention times.

GAS CHROMATOGRAPHY




injector
detector
N_2
oven

PRACTICAL REQUIREMENTS

Carrier gas

- Flow regulators & Flow meters
- **Injection devices**
- **Columns**
- **Temperature control devices**
- **Detectors**
- **Recorders & Integrators**

Requirements of a carrier gas

- Inertness
- Suitable for the detector
- High purity
- Easily available
- Cheap
- Should not cause the risk of fire
- Should give best column performance

How to select a Carrier gas

Depending on	**priority**
• Availability	• first
• Purity	• Second
• Coast	• Third
• Type of Detector	• Fourth
• consumption	• Fifth

Required Gases Purities

- ***Helium*** For carrier gas: 99.995% high purity, with less than 1.0 ppm each of
- water, oxygen, and total hydrocarbons after purification.
- Use water, oxygen, and hydrocarbon traps.
- ***Hydrogen*** For carrier or detector fuel gas: 99.995% high purity, with <
- 1.0 ppm of total hydrocarbons after purification.
- Use water, oxygen and hydrocarbon traps.

Required Gases Purities

Air For detector fuel gas: 99.995% high purity. •

• Air compressors are not acceptable because • they do not

• meet pressure, water, and hydrocarbon • requirements.

Nitrogen For carrier or make-up gas: 99.995% • high purity, with less than 1.0

• ppm of total hydrocarbons after purification. •

Argon 5% Methane For ECD make-up gas: • 99.995% high purity.

Carrier Gas Control

The Flow mode has four options for the carrier •
gas control: •

- Constant flow •
- Constant pressure •
- Programmed flow •
- Programmed pressure •

Flow regulators & Flow meters

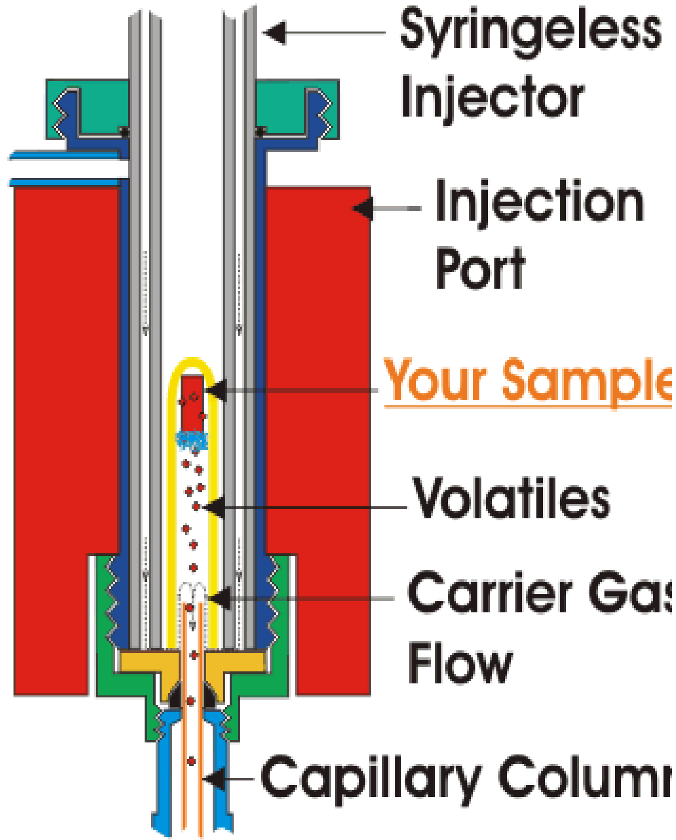
X□ deliver the gas with uniform
pressure/flow
rate

X□ **flow meters:-** Rota meter & Soap
bubble
flow meter

Injection Devices

Gases can be introduced into the column by valve devices

□ liquids can be injected through loop or septum devices



COLUMNS

- Important part of GC
- Made up of glass or stainless steel
- Glass column- inert , highly fragile

COLUMNS can be classified

□ Depending on its use

1. Analytical column

1-1.5 meters length & 3-6 mm d.m

2. Preparative column

3-6 meters length, 6-9mm d.m

Depending on its nature

1.Packed column: columns are available in a packed manner

S.P for GLC: polyethylene glycol, esters, amides, hydrocarbons, polysiloxanes…

2.Open tubular or Capillary column or Golay column

- Long capillary tubing 30-90 M in length
- Uniform & narrow d.m of 0.025 - 0.075 cm
- Made up of stainless steel & form of a coil
- Disadvantage: more sample cannot loaded

2. Column

The column •

• **Is where the chromatographic separation • of the sample occurs. •**

• Several types of columns are available for • different chromatographic applications: •

• The heart of the system. •

• It is coated with a stationary phase which • greatly influences the separation of the • compounds. •

Factors Affecting Column Separations

Volatility of compound: Low boiling (volatile) •
components will travel faster through the
column than will high boiling components •

• **Polarity of compounds**: Polar compounds •
will move more slowly, especially if the column is
polar. •

• **Column temperature**: Raising the column •
temperature speeds up All the compounds in a
mixture, “Columns have lower and upper •
temperature limits”.

Factors Affecting Column Separations

- **Column packing polarity**: Usually, all compounds will move slower on polar columns, but
- polar compounds will show a larger effect.
- **Flow rate of the gas through the column**:
- Speeding up the carrier gas flow increases the
- speed with which all compounds move through the column.
- **Length of the column**: The longer the column, the longer it will take all compounds to elute.
- Longer columns are employed to obtain better separation.

GLC

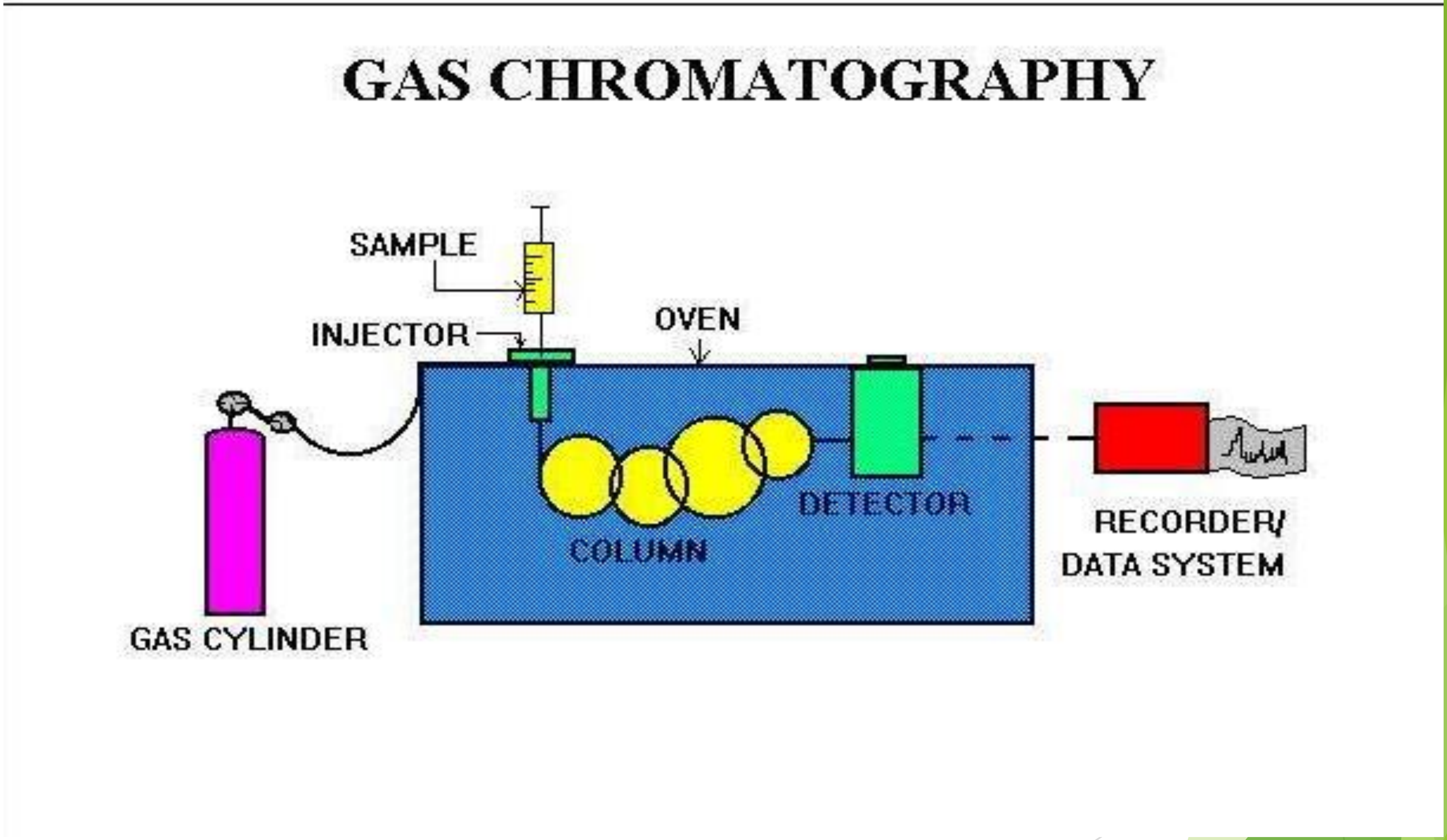
Carrier gas

- Flow regulators & Flow meters
- **Injection devices**
- **Columns**
- **Temperature control devices**

- **Detectors**
- **Recorders & Integrators**

GC Part (2)

Gas Liquid Chromatography



GAS CHROMATOGRAPHY
SAMPLE
INJECTOR
OVEN
GAS CYLINDER
COLUMN
DETECTOR
RECORDER/
DATA SYSTEM

GC

Carrier gas

- Flow regulators & Flow meters
- **Injection devices**
- **Columns**
- **Temperature control devices**

- **Detectors**
- **Recorders & Integrators**

Detector

- **The part of a gas chromatograph**
- **which signals the change in**
- **composition of the mixture passing**
- **through it.**

Detector types

- 1. Electron Capture Detector.
- 2. Flame ionization Detector.
- 3. Nitrogen Phosphors Detector.
- 4. Thermal Conductivity Detector.

Detector types

- 5. Flame Photometric Detector.
- 6. Photo ionization Detector.
- 7. Electrolytic Conductivity Detector.
- 8. Mass Spectrometric Detector.

1. Electron Capture Detector (ECD)

- **Mechanism:**
- • Electrons are supplied from a 63Ni foil lining the detector cell. A current is generated in the cell
- .Electronegative compounds capture electrons resulting in a reduction in the current.
- The amount of current loss is indirectly measured and a signal is generated.

ECD

- • **Selectivity**: Halogens, nitrates, conjugated carbonyls
- • **Sensitivity**: 0.1-10 pg (halogenated compounds);
- 1-100 pg (nitrates); 0.1-1 ng (carbonyls)
- Linear range: 1000-10000
- Gases: Nitrogen or argon/methane
- Temperature: 300-400°C

2. Flame ionization Detector (FID)

- **Mechanism:**
- • Compounds are burned in a hydrogen-air flame.
- • Carbon containing compounds produce ions that are attracted to the collector.
- • The No. of ions hitting the collector is measured and a signal is generated.

FID

- **Selectivity**: Compounds with C-H bonds.
- • **Sensitivity**: 0.1-10 ng
- Gases: Combustion hydrogen and air; Makeup He or N2
- Temperature: 250-300°C,and 400-450°C for high temp.

3. Nitrogen Phosphors Detector (NPD)

- **Mechanism:**
- • Compounds are burned • Nitrogen and phosphorous containing compounds
- produce ions that are attracted to the collector.
- • The number of ions hitting the collector is measured and a signal is generated.

NPD

- **Selectivity**: Nitrogen and phosphorous
- • **Sensitivity**: 1-10 pg
- Gases: Combustion - hydrogen and air; Makeup - Helium
- Temperature: 250-300°C

4. Thermal Conductivity Detector (TCD)

- **Mechanism:**
- • A detector cell contains a heated filament with an applied current.
- As carrier gas containing solutes passes through the cell, a change in the filament current occurs.
- • The current change is compared against the current in a reference cell.
- • The difference is measured and a signal is generated.

works by having two parallel tubes both containing gas and heating coils. The gases are examined by comparing the heat loss rate from the heating coils into the gas. Normally one tube holds a reference gas and the sample to be tested is passed through the other. Using this principle, a TCD senses the changes in the thermal conductivity of the column effluent and compares it to a reference flow of carrier gas. Most compounds have a thermal conductivity much less than that of the common carrier gases of hydrogen or helium.

TCD

- • **Selectivity:** All compounds except for the carrier gas
- • **Sensitivity:** 5-20 ng
- Linear range: 105 -106
- Gases: Makeup - same as the carrier gas
- Temperature: 150-250°C

5. Flame Photometric Detector (FPD)

- **Mechanism:**
- • Compounds are burned in a hydrogen-air flame.
- Sulfur and phosphorous containing compounds produce light emitting species (sulfur at 394 nm and phosphorous at 526 nm). A
- monochromatic filter allows only one of the wavelengths to pass. A photomultiplier tube is used to measure the amount of light and a signal is generated.
- A different filter is required for each detection mode.

FPD

- **Selectivity**: Sulfur or phosphorous containing compounds.
- • **Sensitivity**: 10-100 pg (sulfur); 1-10 pg (phosphorous)
- Linear range: Non-linear (sulfur); 103 -105 (phosphorous)
- Gases: Combustion - hydrogen and air; Makeup - nitrogen
- Temperature: 250-300°C

6. Photo ionization Detector (PID)

- **Mechanism:**
- • Compounds eluting into a cell are bombarded with high energy photons emitted from a lamp.
- Compounds with ionization potentials below the photon energy are ionized.
- The resulting ions are attracted to an electrode, measured, and a signal is generated.

PID

- • **Selectivity**: Depends on lamp energy. Usually used for aromatics and olefins (10 eV lamp).
- • **Sensitivity**: 25-50 pg (aromatics); 50-200 pg (olefins)
- Linear range: 105 -106
- Gases: Makeup - same as the carrier gas
- Temperature: 200°C

7. Electrolytic Conductivity Detector *(ELCD)*

- **Mechanism:**
- • Compounds are mixed with a reaction gas and passed through a high temperature reaction tube.
- Specific reaction products are created which mix with a solvent and pass through an electrolytic
- conductivity cell. The change in the electrolytic conductivity of the solvent is measured and a
- signal is generated. Reaction tube temperature and solvent determine which types of compounds are detected.

ELCD

- **Selectivity**: Halogens, sulfur or nitrogen containing compounds.
- • **Sensitivity**: 5-10 pg (halogens); 10-20 pg (S); 10-20 pg (N)
- Linear range: 105 -106 (halogens); 104 -105 (N); 103.5-104(S)
- Gases: Hydrogen (halogens and nitrogen); air (sulfur)
- Temperature: 800-1000°C (halogens), 850-925°C (N), 750-825°C (S)

8. Mass Detector (MS)

- **Mechanism:**
- • Compounds are bombarded with electrons (EI) or gas molecules (CI). then fragmented into characteristic
- charged ions or fragments. The resulting ions are focused and accelerated into a mass filter.
- mass filter selectively allows all ions of a specific mass to pass through to the electron multiplier. All of the ions of the
- specific mass are detected. The mass filter then allows the next mass to pass

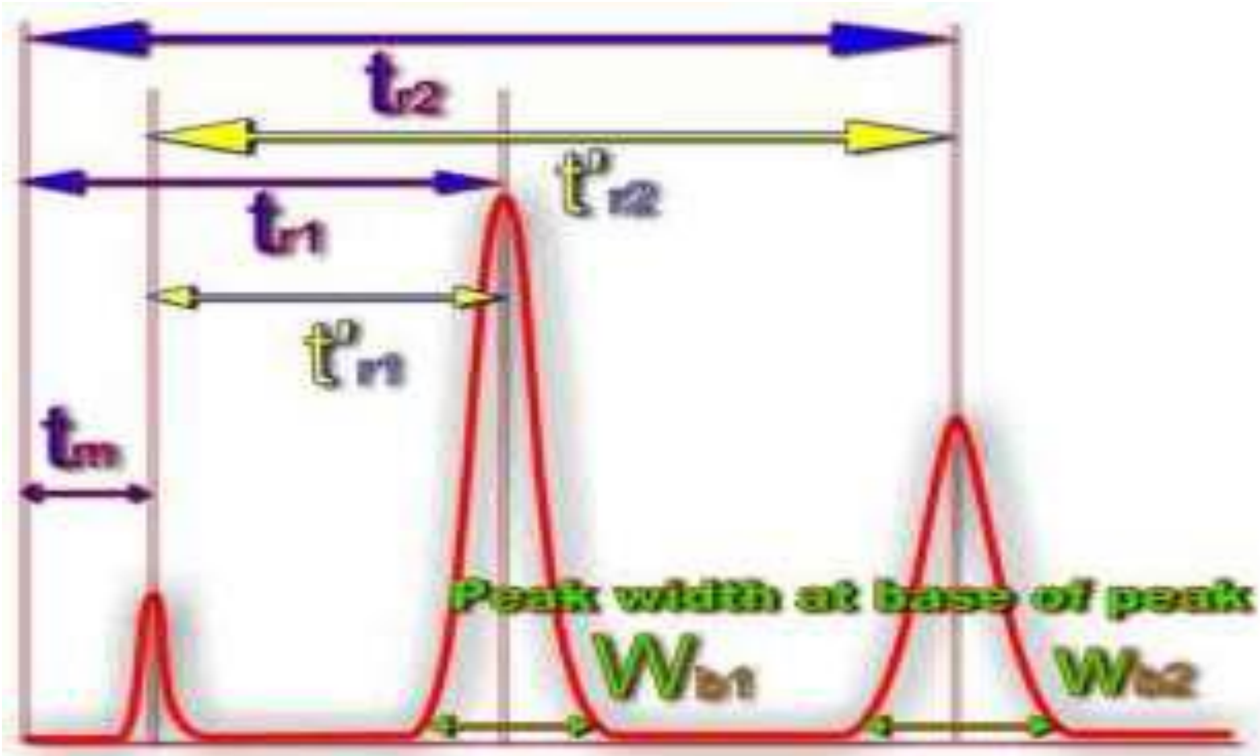
Good Detector

- 1.High sensitivity.
- 2. Rapidly respond to concentration changes.
- 3. Large linear range.
- 4. Stable with respect to noise and drift.
- 5. Low sensitivity to variation in flow,.
- 6. Possible selectivity.
- 8. Produces an easily handled signal.
- 9. A temperature range from room temperature to at least 400 C

Chromatogram

*The data recorder **plots the signal from the detector over time.**

- The retention time, is qualitatively indicative of the type of compound.
- The area under the peaks or the height of the peak is indicative of the amount of each component



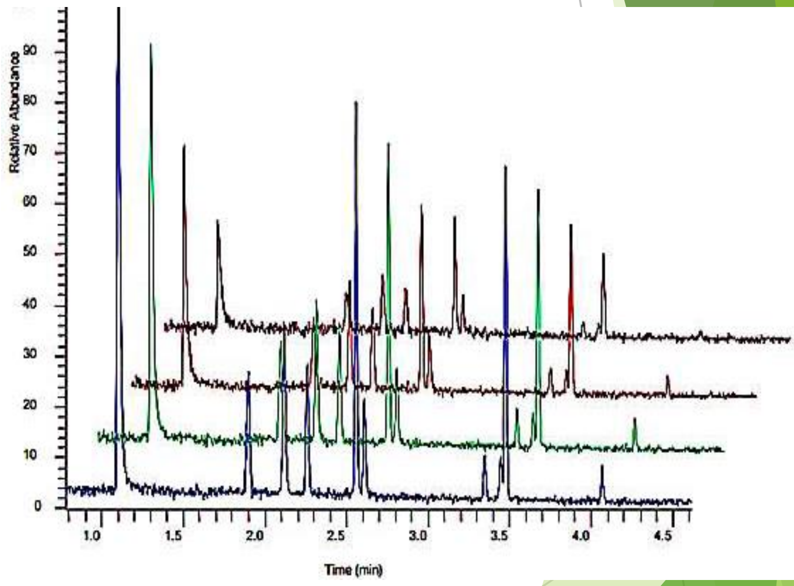
t r2
t' r2
t r1
t' r1
t m
Peak width at base of peak
W b1
W b2

Retention Time (RT)

RT

- **RT, is the time it takes for a compound to travel**
- **from the injection port to the detector.**
- • Thousands of chemicals may have the same
- retention time, peak shape, and detector
- response.
- • For example, under certain conditions, DDT has
- the same retention time as PCBs
- (polychlorinated biphenyls).

Chromatogram



Applications

- the environmental
- • Testing or commercial laboratories
- • Industrial laboratories
- • Government laboratories
- • Research institutes
- **Petrochemical and Gas**
- • Refinery
- • Oil Industry
- • Gas suppliers

applications

- **Clean water analysis**
- **Pollutants in water**
- • Halocarbons
- • Acid priority pollutants: phenols,
- chlorophenols, nitrophenols
- • Pesticides
- **Other fields such as:** pharmaceutical
- foods