

(2015)

Q #2(i)

Differentiate b/w

Normal phase chromatography:- Reverse phase chromatography:-

- \* A separation method which allows the distribution of components of a mixture b/w two phases, one of which is polar stationary phase while the mobile phase is non-polar.
- \* Evolved in the 1970s, in the form of liquid.
- \* Uses a polar stationary phase which is mainly a pure silica.
- \* The mobile phase carries non-polar analytes at the beginning of separation. Has a poor reproducibility of the retention time.
- The separation method more polar than stationary phase.
- A recently evolved form of HPLC.
- Uses a non-polar stationary phase, which is a modified silica substrate with long hydrophobic chains.
- The mobile phase carries polar analyte.
- Has a higher reproducibility of the retention time.

(ii) Advantages of gradient elusion over isocratic elusion:-

Gradient elusion:-

- \* Mobile phase solvent composition increases with time.

- \* Best for analysis of complex samples.

- \* Often used in method development for unknown mixtures.

- \* Increases sensitivity of analysis.

Iso-cratic elusion:-

Mobile phase solvent composition remains const with time.

Best for simple separations.

Often used in quality control applications that support and are in close approximilay to a manufacturing process.

Sensitivity of analysis not increases as compared to gradient elusion.

(iii) Why it is preferred compared to packed

Packed columns:-

- \* A column that contains a fully packed stationary phase made up of fine particles.

- \* Have a low efficiency

to use capillary columns

columns:-

Capillary columns:-  
A column whose stationary phase is coated on the inner surface.

Have a higher efficiency.

- |   |  |
|---|--|
| * Requires a large amount of sample.    | Require only a small amount of the sample. |
| * Have high pressure inside the column. | Have less pressure inside the column.      |
| * Less expensive.                       | More expensive.                            |

(iv)

### Selection criteria for a gas in GC?

- \* Stationary phase:-

They are close to polarity of solutes.

- \* Columns diameter:-

Have small diameter (0.25mm) when sample overloaded is not a problem.

- \* Film thickness:-

- \* If thin then high boiling point solutes.
- \* If thick then low boiling point solutes.

- \* Column lengths:

30mts → Most application.

15mts → For simple samples

60mts → For complex samples.

(v)

### Function of programmer in thermobalance:-

A wide variety of fully electronic temperature controller and programmener are available commercially.

The basic requirements of a balance are accuracy, sensitivity, reproducibility and capacity. Temperature sensors are either platinum resistance thermometers or thermo-couples. Heating rates often range from fraction of a degree per minute to nearly  $1000^{\circ}\text{C}$  per minutes with facilities for iso-thermal operation. In a small thermal analysis laboratory one programmer may serve several instruments.

(i)

Factors effecting DTA curve:-

- \* Many of the factors are associated with instrumental designs, sample characteristics include following.
- \* Amount of sample + Particle size
- \* Packing density. \* Heat capacity.
- \* Thermal conductivity \* Degree of crystallinity
- \* Diluent \* Swelling and shrinkage.

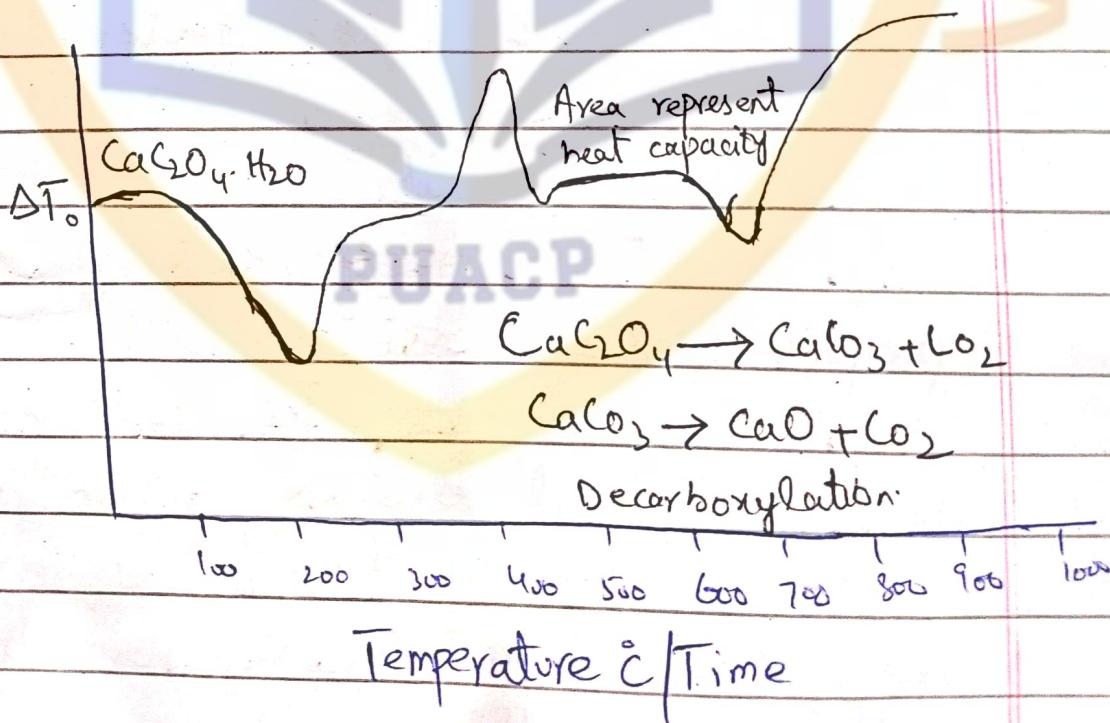
(ii)

What type of information obtained from DSC curve?

DSC measures the

energy absorbed or released by a sample as it is heated or cooled. DSC curve of a pure compound represent the characteristics of that compound for physical and chemical changes. Using DSC curve one can correlate the changes in energy because of thermo-physical and chemical changes occurring in a compound because of heating.

Change in DSC curve also gives us information like melting point, glass transition temperature, crystallization temperature etc. The DSC curve of calcium oxalate ( $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ ).



(viii)

Justify that KCl is the ideal electrolyte for salt bridges.

Potassium chloride (KCl)

is the ideal species for incorporation into a salt bridge as  $K^+$  and  $Cl^-$  have the same number of electrons and are approximately the same size. As we know that charges flow due to presence of electric field. An salt bridge over all bridge electric field is existed due to electric field of ions and electrodes. With this principle KCl is used to maintain neutrality in the solution containing <sup>suitable</sup> solution so reaction is not reached equilibrium as faster.

\* Liquid  
b/w  
electro  
electr  
  
A j  
liquid  
Cause  
and

(ix)

Why saturated calomel electrode is preferred over S.H.E.:-

The potential of standard calomel electrode containing an on-saturated solution of KCl is less temperature dependent. But its potential will change with the change in concentration of chloride ions and change in activity of chloride ions. So,

it is preferred over SHE.

(X)

- \* Liquid junction potential in potentiometry:-  
Liquid junction interface  
b/w two solutions containing different  
electrolytes or different of same  
electrolyte.
- \* A junction potential occurs at every  
liquid junction.
- \* Caused by unequal mobilities of +ve  
and -ve ions

2016

Q # 2(i)

## Thermal analysis:-

<sup>66</sup> Thermal analysis is a branch of materials science where the properties of materials are studied as they change with temperature.

Several methods are commonly used. They are distinguished from one another by the property which is measured.

It depends on physical properties of certain substance.

Physical properties include mass, temperature, enthalpy etc. These are techniques of thermal analysis.

## Glass transition temperature:-

Glass transition may occur as the temperature of an amorphous solid is increased. This transition appears as a step in the baseline of the recorded DSC signal.

This is due to the sample undergoing a change in heat capacity, no formal phase change occurs.

(iii)

### Applications of DSC:-

Following are some applications;

- \* Composition of polymers is determined.
- \* Melting and glass transition temperature can be determined.
- \* This can show the polymer degradation by lowering the M.P. (molecular weight & low melting point) can be determined.
- \* Thermal degradation of impurities.
- \* Compare additive effects on material.

(iv)

### Resolution in HPLC:-

The resolution of elusion is a quantitative measure of how will two elusion peaks can be differentiated in chromatographic separation.

It can be defined as "ability to separate two signals i.e separation of two constituents."

Higher the resolution the easier it is to achieve base line separation b/w two peaks.

Differentiate b/w V gradient and iso-catic elusion:-  
Already done #

(vi)  
Characteristics of detector in HPLC:-

- Ideal detectors have following properties;
- \* High sensitivity
  - \* Low dead value
  - \* Give large linear response range.
  - \* It is non-destructive
  - \* Reliable and easy to use.

(vii)

Why deactivation is necessary in GC?

"Deactivation is the process of chemically modifying a compound to produce new compound which has properties that are suitable for analysis using a GC?"

The chemical structure of the compound remains the same and just modifies the special functional groups of reacting compound to derive ~~derivative~~ chemical and physical properties in order

to make them detectable and analysable.

Deactivation also reduces analyte adsorption in GC system and improves detector response, peak separation and peak symmetry.

PLC:-

ing

value

(viii)

Compare

Thermal conductivity:-

It is a simple detector.

General response to both organic and inorganic species.

Large linear dynamic range ( $10^5$ )

Non destructive, solutes can be collected after detection.

Flame ionization:-

Universal detector for organics.

Do not respond to common inorganic compounds.

Linear and dynamic range better than TCD.

Carrier gas not detected.

(ix)

Ion selective electrode:-

Advantages:-

These type of electrodes are unaffected by

Disadvantages:-

Electrodes are fragile and have

sample colour or

Limited shelf life

turbidity

- \* Non destructive or no consumption of analyte

- \* Non contaminating.

Short response time

in sec or mn useful

in industrial applications

- \* ISEs are one of few techniques which to the activity of can be measure both positive and negative ions.

Electrodes can be fouled by proteins or other organic solutes

Electrodes respond to the activity of un-complexed ions, ligands must be absent.

the electrode has finds of h

(\*)

How electron capture detector works:-

The ECD uses a radioactive

$\beta$  emitter (electrons) to ionize some of the carrier gas and produces a current b/w a bound pair of electrodes

When organic molecules that contain electronegative functional groups such as halogens, phosphorous and nitro groups pass by the detector, they capture some of the electrons and reduce

balance analysis

involve

changes

transf

Evolv

dete

the

samp

the ~~current~~ current measured b/w electrodes. The ECD is as sensitive as the FID (Flame ionization detector) but has a limited dynamic range and finds its greatest application in analysis of halogenated compounds.

2017

Q # 2(i)

Thermobalance:-

In analytical chemistry a balance is used for thermogravimetric analysis known as thermobalance. It involves the measurement of weight changes associated with the transformation of matter ~~with~~ when heated.

(ii)

Evolved gas detector:-

Evolved gas detector is a method used to study the gas evolved from a heated sample that undergoes decomposition.

or desorption. It is either possible just to detect evolved gases using EGD or to analyse explicitly when gases evolved using evolved gas analysis (EGA).

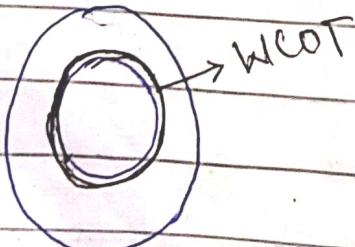
### WCOT:-

- \* Wall coated open tubular column was first introduced by Golay in 1957.

### In LCOT column

wall is directly coated with the stationary phase layer at a film thickness of 0.05-3 μm.

- \* WCOT have low sample capacity.



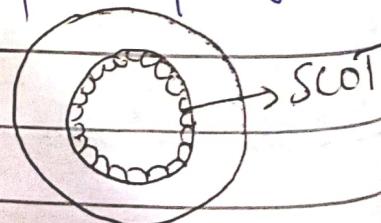
(iii)

### SCOT:-

- \* Support coated open tubular column was first introduced by Holzä and Harvath in 1963.

These columns contain an absorbed layer of a very fine solid support coated with the liquid phase.

SCOT columns can hold more liquid phase and have higher sample capacity.



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DTA

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(iv)

Difference b/w

DTA:-

\* It stands for differential thermal analysis.

\* The temperature difference developed b/w a sample and a reference compound is increased at identical heat measurements.

\* Used to analyse the thermal properties of minerals for the characterization of polymers and biological materials.

\* Sample can be used as a solid substances.

DSC:-

It stands for differential scanning calorimetry.

The heat flow is measured against the temperature change at a particular time.

Used to analyse proteins and antibodies etc.

Sample is always liquid.

(v)

Pre-columns in HPLC:-

A pre-column is also called guard column protects the main column of plugging and contamination by samples and mobile phase. In other words it is a

protective column or cartridge controlled b/w the injector and analytical column. It serves to remove the impurities and suspended solids from reacting the analytical column.

(vi)

Electrode potential:-

Electrode potential

is the electric potential in an electric component. In a cell, there is an electrode potential for the cathode and electrode potential for the anode. The difference b/w the two electrode potential equals to cell potential.

$$E_{\text{cell}} = E_{\text{cathode}} - E_{\text{anode}}$$

(vii)

Nernst equation:-

working

Nernst made a quantitative relationship b/w equilibrium cell potential and concentration and equation deduced by him;

$$E_{\text{cell}} = E_{\text{cell}}^{\circ} - \frac{RT}{nF} \log K$$

where,

$E_{\text{cell}}^{\circ}$  = standard cell potential.

R = Universal gas constant.

$n$  = No. of ions.

$f$  = Faraday

$\log K = K$  at equilibrium.

It is very helpful in determining cell potential and equilibrium constant.

(VIII)

Stepwise elusion:-

"Process in which composition of mobile phase is changed in steps during a single chromatographic run."

Stepwise elusion (SE) can work as a method for the purification and concentration of proteins at the same time. The concentration factors of proteins by SE with ion exchange chromatography columns were measured as a function of the feed volume and feed concentration.

(IX)

Standard columns in HPLC:-

The most important part of HPLC system is column which is made up of glass.

plastic or stainless steel. Mostly stainless steel is used to withstand high pressure as well as chemical action of solvents. Mostly 2 columns are used in HPLC.

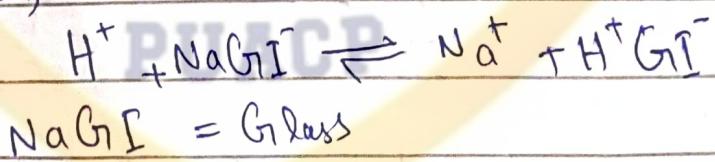
• Guard column \* Analytical column.

Guard column contain same stationary phase as the main column, so protects the main column, extends the life of main column.

Analytical columns have large size than good column and have more stationary b/w them.

Equation for pH measurement in glass electrodes:-

The ion exchange reaction can be written as;



$\text{H}\text{G}\bar{\text{I}}$  = Silicic acid.

where  $\text{G}\bar{\text{I}}$  represents many very charged sites of glass surface. The equilibrium constant for this purpose is so large that the surface of glass membrane mainly consist of silicic acid ( $\text{H}^+\text{G}\bar{\text{I}}^-$ ).

(2018)

Q # 2(i)

Thermocouple:-

A thermocouple is a temperature measuring device and based on the principle that two wires made of dissimilar conductive materials connected at either end will generate a potential b/w the two ends that is a function of materials and temperature difference b/w two ends. Thermocouple have following functions.

- \* If offers good reproducibility.
  - \* Have high speed of response
- Comparatively cheaper in cost.

(ii)

Difference b/w DSC and DTG:-

Already done #

(iii)

Electrode potential:-

Already done #

(iv)

Composition of glass membrane in  
glass electrodes:-

The composition of glass membrane  
is given in the following.

- \* It is composed of 72% silicon dioxide ( $\text{SiO}_2$ )
- \* It is composed of 22%  $\text{Na}_2\text{O}$ .
- \* 6%  $\text{CaO}$

The most important advantage is that  
these membranes are superior, selective  
and have much better life time.

(v)

Three types of open tubular columns in  
GC:-

Following are three main types;

- i) Porous layer open tubular column (PLOT)
- ii) Wall coated open tubular column (WCOT)
- iii) Support coated open tubular column (SCOT)

PLOT:-

Inner layer is coated with solid  
stationary phase particles. Plot does  
not have a liquid stationary phase.

SCOT:-

Inner layer is of solid support  
coated with liquid stationary phase

Open to  
amount

WCOT:

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and

Differ  
GSC:-

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GSC  
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GSC  
column

Open tubular column has a great amount of S.P than the WCOT.

WCOT:-

Inner coated with the liquid stationary phase (S.P). Easy to use and flexible

(vi)

Differentiate b/w

GSC:-

GLC:-

- |  |  |
|--|--|
| * In GSC, the stationary phase is in the solid state.                  | In GLC, the stationary phase is in liquid state                        |
| * GSC is an adsorption chromatographic technique.                      | GLC is a partition chromatography technique                            |
| * In GSC, the distribution co-efficients of compounds are much higher. | In GLC, the distribution co-efficients are comparatively much smaller. |
| * The retention time ( $R_f$ ) is comparatively long in GSC.           | The retention time is comparatively short in GLC.                      |
| * GSC uses packed columns.   | GLC uses capillary columns.  |

(vii)

Eddy diffusion:-

"Effect of different path lengths due to irregular flow molecules through packed particles in a column."

Each molecule see different paths causing the solute molecules to arrive at the column outlet at different times.

It is independent of mobile phase velocity.

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phase  
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Adsor

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(viii)

Van Deemter equation:-

The Van Deemter

equation is the relationship b/w height equivalent to a theoretical plate (HETP) which is a measure of column efficiency and linear velocity  $v$

$$HETP = \frac{A \cdot B}{v} + (C_s + C_m) v \quad (\text{signs clear})$$

HETP = Height equivalent to theoretical plate  
The terms involved;

A is Eddy diffusion.

B is longitudinal diffusion co-efficient

C<sub>m</sub> = mass transfer co-efficient

Pre

C<sub>s</sub> is divided into C<sub>s</sub> and C<sub>m</sub> contribution  
from stationary phase and mobile  
phase M.P respectively.  
 $\mu$  is linear velocity.

(ix)

Adsorbents used in HPLC columns:-

Adsorbent:-

- \* Should be inert (should not react with eluting solvent).
- \* Should be stable for long period.
- \* Should be cheaper.

Most commonly used adsorbents are;

Silica gel → AA and steriods.

Alumina → small organic molecules.

Activated carbon - Carbohydrates and proteins.

Most strongly adsorb component forms top most band while least adsorb component forms lower most band on adsorbent media.

(x)

Pre-columns:-

Already done #

(2019)

Q # 2(i)

Advantages of thermal conductivity:-

The

advantages of thermal conductivity detectors  
are;

- \* General response to both organic and inorganic species.
- \* Have a non-destructive character.
- \* It permits the re-use of solution after detection.
- \* Large linear dynamic range  $\approx 10^5$ .
- \* Have a high sensitivity.

(ii)

Temperature programming:-

In gas chromatography, it is often difficult to resolve components completely without using a technique known as temperature programming. It changes the retention time compared to a isothermal run but if the same temperature <sup>is kept</sup> is used elution times will remain

constant for each component. temp programming is usually applied to samples containing a mixture of ~~components~~ components that have boiling point within a narrow range. Temperature programming keeps the temperature of a GC column at a fixed volume as the separation proceeds.

(iii)

Disadvantages of H<sub>2</sub> gas:-

- \* It is a reductive gas.
- \* Hydrogen can form an explosive mixture with air.

Some industries in some countries have regulated against the use of hydrogen.

- \* Hydrogen is cheap and can be easily and readily made from H<sub>2</sub>O if you have a suitable hydrogen generator.

However hydrogen is flammable and explosive, so it something ever gas wrong.

- \* It reacts with un-saturated compounds and is flammable.

(iv)

## Retention time:-

Retention time is

a measure of time taken for solute to pass through chromatography column. It is calculated as the time from injection to detection.

The  $R_f$  for a compound is not fixed as many factors can influence it even if the same GC and column are used these include the gas flow rate.

Factors:-

It depends on many factors

- \* Analysis conditions      \* Types of columns
- \* Degradation of columns      \* Existence of active point such as contamination.

(v)

## Principle of DTA:-

The basic principle involved in DTA is the temperature difference (DT) b/w test sample and an inert reference sample under controlled and identical conditions of heating or cooling is recorded continuously as a function of temperature.

or time, thus the heat absorbed or emitted by a chemical system is determined.

Principle of TGA:-

In TGA, the sample is heated in a given environment (air,  $N_2$ ,  $CO_2$ , Ar, He etc) at controlled rate. The change in the weight of the substance is recorded as a function of temperature or time. The temperature is increased at constant rate of known initial weight of the substance and the changes in weights are recorded as a function of temperature at different time interval.

This plot of weight change against temperature is called TGA.

(vi)

Differentiate b/w normal phase and reverse phase chromatography:-

Already done #

(vii)

Role of liquid junction potential in potentiometry:-

Already done #

OR

(viii)

## Characteristics of ideal reference electrodes:-

A reference electrode is an electrode that has the half cell potential known constant and completely determined to the composition of the solution under study.

In conjunction with this reference is the indicator or working electrode whose response depends upon the analyte concentration.\* Basic function of a reference electrode is to maintain a constant electric potential against which deviations may be measured.

(ix)

## Potentiometric titrations:-

In potentiometric titration the equivalence point is determined by the measurement of an indicator electrode potential against a convenient reference electrode and plotting the change of the potential difference against volume of titrant is called potentiometric titration.

(X)

## DTA Curve:-

DTA is a thermoanalytic technique that is similar to DSC.

DTA curve ~~provides~~ provides data on the transformation that have occurred such as glass transition crystallization melting and sublimation.

## Characteristics of DTA curves:-

### DTA curves

are not only help on the identification of materials but their peaks provide quantitative information regarding mass of sample, heat of reactions (enthalpy change) and factor such as sample geometry and thermal conductivity.

If latter two factors are expressed by a factor ( $K$ ) called calibration factors then peak can be expressed as

$$\text{Peak} = \pm \Delta H$$

Incomplete

Diagram

(2020)

Q # 2 (i)

Advantages of HPLC:-

⇒ Short analysis time

⇒ HPLC was developed in the mid 1970s in which high pressure pumps were employed to improve eluent flow.

⇒ Column with small uniform particles, for fast and high resolution separation of compounds.

⇒ This is more powerful separation technique than all other chromatographic techniques which is used to separate and identify organic, inorganic, biological and pharmaceutical products as well.

\* Higher efficiency, \* Rapid separation

Q # 2 (ii)

Flame ionization detector:-

It is the most frequently used detector in GC.

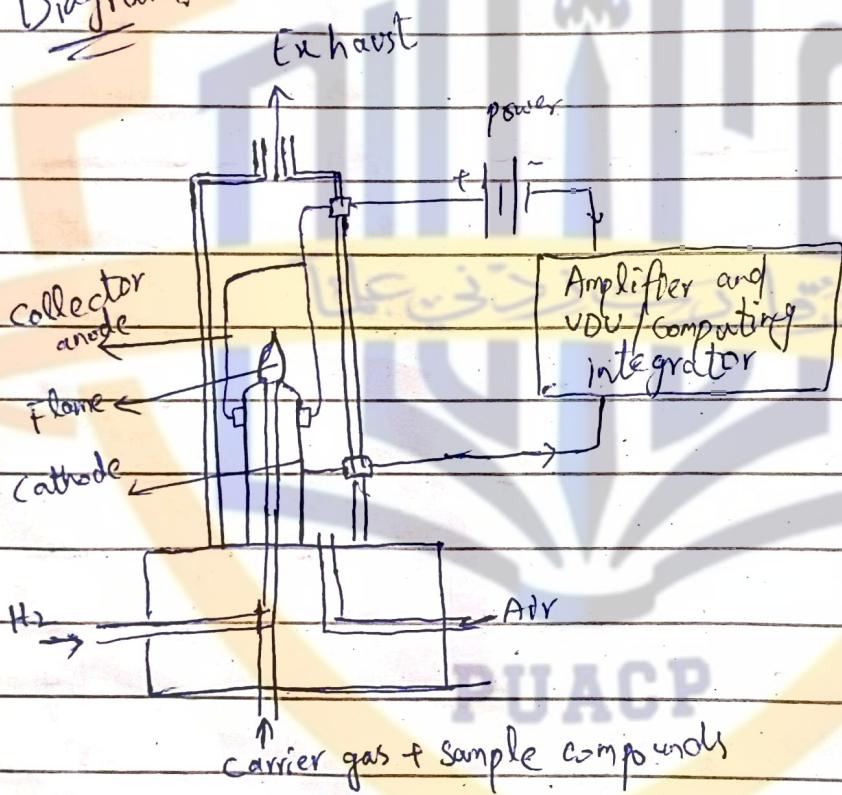
Working:-

Effluent gas from the column is mixed with hydrogen and air and burned at a small metal jet. The jet forms the negative electrode of an electrolytic cell, the positive or collector electrode

being a loop of wire or short tube placed just above the flame. The potential difference applied across the electrodes is about 200V.

The FID responds to virtually all organic compounds except formic acid, air and other inorganic species. Its response to water is very low. It has a high sensitivity and the widest linear range ( $10^7$ ) of any detector in common use.

Diagram:



Q # 2(vii)

Advantages of  $\text{Ag}/\text{AgCl}$ :

~~sample~~  
many features making it suitable for  
use in the field:

The electrode has

- \* Simple construction      \* Inexpensive to manufacture.
- \* Stable potential      \* Non-toxic components.

They are usually manufactured with saturated KCl electrolyte, but can be used with lower concentrations such as 1 mole/kg KCl.

### Disadvantages:-

It has some disadvantages; change in the electrolyte concentration will change the electrode potential.

Silver chloride is slightly soluble in strong KCl solution, so it is sometimes recommended to saturate the KCl with AgCl to avoid stripping the silver chloride off the silver wire.



Q#2(w)

### Gradient elusion:

Gradient elusion can separate solutes that can't be separated by isocratic operation. It is especially effective where the sample components differ widely in polarity.

### Procedure:

For gradient elusion, the solvents from separate reservoirs are fed to a mixing chamber and the mixed

solvent is then pumped to the column. Many commercial chromatographs have systems, that can mix two or more solvents in a progressive manner from 0 to 100% of one component so that a solvent composition profile is generated which enable separation of closely eluting peaks in one part of the chromatogram without changing the resolution in other parts.

### Limitations:

There are several constraints on this technique.

- \* If one of the solvent give an appreciable response at the detector (say absorption at a UV detector) then the generation of a solvent gradient will also introduce a baseline drift in response.
- \* The column will also need time to regenerate to the starting solvent composition each time a fresh gradient run is started.
- \* The selection of correct solvent connected to the injection part of the chromatograph and purged with carrier gas whilst being rapidly heated. This causes any previous adsorbed substance to be thermally

described and swept onto the column in a narrow band to be separated in the normal way.

### Q # 2uv)

UV detectors used in HPLC:-

Most widely

Ackai

used (about 80%) HPLC detectors are UV detectors. UV absorption detector works by measuring UV absorption of effluent from the column as it passes through a small flow cell held in the radiation beam. The UV detector has much better sensitivity about  $10^{-8}$  g/ml ( $0.01\text{ppm}$ ). It is not temperature sensitive. It is generally suitable for gradient elusion work because many of the solvents used in HPLC do not absorb to any significant extent at the wavelengths used for monitoring the column effluent. Both single and double beam instruments are commercially available.

Various designs of U.V detectors:-

i) Fixed wavelength (Photometer).

ii) Variable wavelength (Spectrophotometer).

iii) Photo diode array detector.

(Q # 2 vii)

Glass transition temperature:-

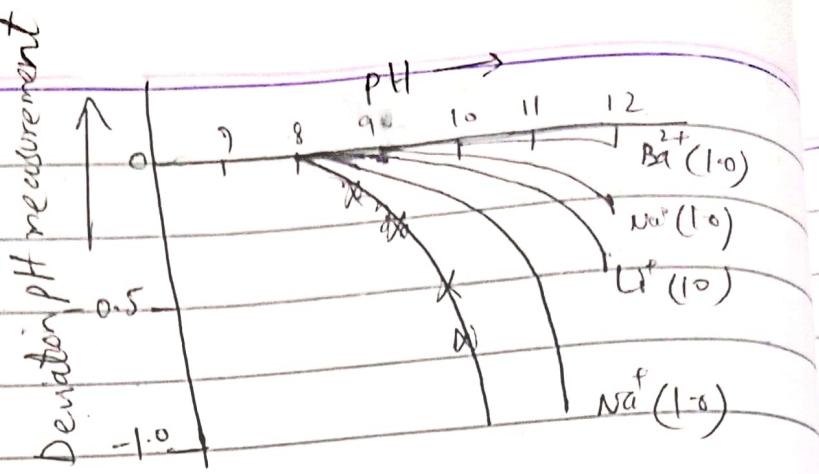
Already done #

(Q # 2 viii)

Alkaline error in potentiometry:-

It is

noticed that the ordinary glass electrode becomes sensitive to alkali ions and gives low reading in high pH range - above 9 or 10 pH units. The reason for the error is that whilst the glass membrane is selective to hydrogen ions, it also responds to other ions. This becomes more significant when the activity of the other ions is higher. Below is a diagram showing the error produced by different cations at different concentrations. You may have noticed that alkaline error is relatively more in case of sodium ion. This is because of higher selective coefficients of sodium ion. This due to sodium ion can be reduced by the use of Li<sub>2</sub>O glass in place of Na<sub>2</sub>O glass.



### G #2 (viii)

Gas sensing probes:

It is a galvanic cell whose potential is related to the concentration of a gas in a solution. In instrument brochures, these devices are often called gas-sensing electrodes, which is a monomer.

Construction

It consists of a tube containing a reference electrode, a specific ion electrode and an electrolyte solution. A thin, replaceable gas-permeable membrane attached to one end of the tube serves as a barrier b/w the internal and analyte solution. As can be seen from figure, this device is a complete electrochemical cell and is more properly referred to as a probe rather than an electrode, a term

that is frequently encountered in advertisements by instrument manufacturers.

Gas-sensing probes are widely used for determining dissolved gases in water and other solvents.

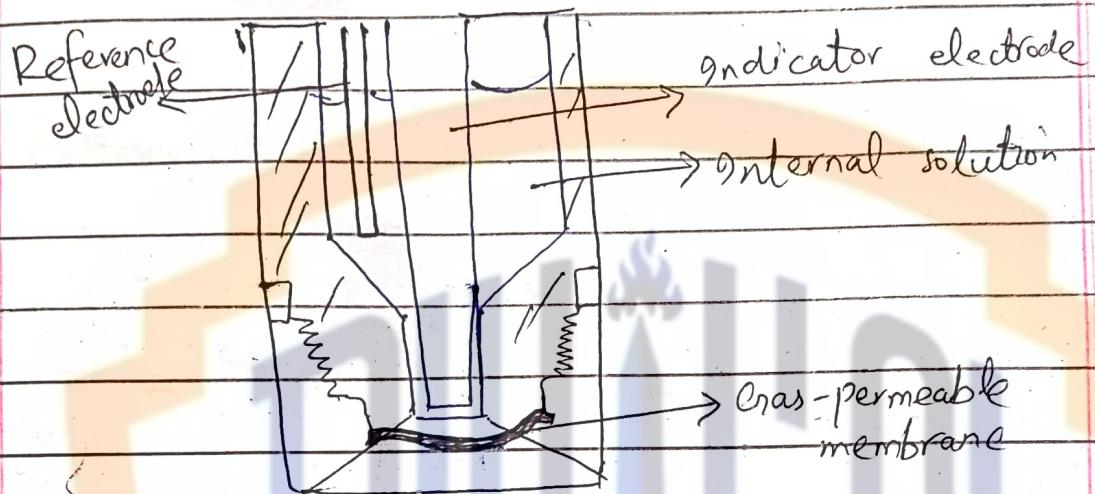


Diagram of gas-sensing probe.

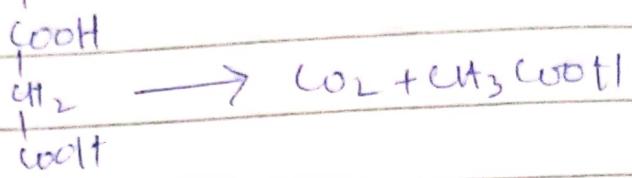
Q # 2 (ix)

How TGA can be used to evaluate the thermal stability of materials?

Many organic compounds have been studied by DTA and TG using the derivatograph. Some interesting examples are given as follows

- i) When the decomposition of malonic acid was studied by DTA and TG techniques, it was found that a phase transition occurred in the region of 70°C with melting

at about  $140^{\circ}\text{C}$  followed by decomposition above  $150^{\circ}\text{C}$ . The process of thermal degradation may be put as:



g<sub>nert</sub>

(b) Another interesting example is the study of glycine by DTA and TG techniques.

The compound melts, with decomposition, in the region of  $220^{\circ}\text{C}$ . During decomposition, the compound glycine foams and it is, therefore, difficult to investigate relatively large samples.

Q # 2 (x)

Basic theory of DTA:-

DTA is a technique in which the temperature of the substance under investigation is compared with the temperature of a thermally inert material such as  $\alpha$ -alumina and is recorded with furnace temperature as the substance heated or cooled at a predetermined uniform rate. The range of temperature measurable in the course of DTA is much larger than TG determination.

(2021)

Q # 2(i)

Inert metallic electrode in potentiometry:-

Several inert conductors respond to redox systems. Such materials as platinum, gold, palladium, and carbon can be used to monitor redox systems. For example, the potential of a platinum electrode immersed in a solution containing cerium (III) and cerium (IV) is

$$E_{\text{ind}} = E_{\text{Ce}^{4+}/\text{Ce}^{3+}}^{\circ} - 0.0592 \log \frac{[\text{Ce}^{4+}]}{[\text{Ce}^{3+}]}$$

A platinum electrode is a convenient indicator electrode for titrations involving standard cerium (IV) solutions.

Q # 2(ii)

Requirements of carrier gas in G.L.C?

The carrier gas used is either helium, nitrogen,  $\text{H}_2$  or organ, the choice of gas depending on factors such as availability, purity required, consumption and the type of detector employed. Thus helium is preferred when thermal conductivity detectors are employed because of its high thermal conductivity relative to that of the

(3)

Vapours of most organic compounds. Associated with this high pressure supply of carrier gas are the attendant pressure regulators and flow meters to control and monitor the carrier gas flow; the operating efficiency of the apparatus is very dependent on the maintenance of a constant flow of carrier gas.

It is appropriate to emphasize here two important safety considerations.

- i) Free-standing gas cylinders must always be supported by means of clamps or chains.
- ii) Waste gases, especially hydrogen, must be vented through an extraction hood.

Q # 2 (iii)

What are the advantages and disadvantages of calomel electrode?

Already done #

Q # 2 (iv)

How preparation of sample is done in thermo gravimetric analysis?

Preparation of sample:-

See Slcoog book TGA

Q #2(v)

Write down the advantages and disadvantages of packed column.

Advantages:

\* The main advantage of packed columns is that in packed columns uniform particle size decreases the multiple bath term in Van Deemter equation.

\* Have high sample capacity & cheaper.

\* Required simple injector system.

\* They are robust.

Disadvantage:

They do not have high resolving power as compared to capillary columns.

\* Less sensitive & Higher/Greater analysis time

Q #2(vi)

Differentiate b/w HPLC and GC?

HPLC:-

| GC:-

i) Definition.

\* HPLC refers to high performance liquid chromatography.      \* GC refers to gas chromatography.

ii) Mobile phase.

\* A solvent in the liquid phase.      \* An inert gas such as helium or nitrogen.

iii) Stationary phase.

\* A solid adsorbent.      \* A liquid on an inert solid support.

(2)

## HPLC

## GC

### (i) Separation

- Applied for separation of fluids.
- Applied for gaseous separations.

### (ii) Temperature control.

- No temperature control is required.
- The column is kept inside an oven to keep the gaseous phase as it is.

### (iii) Column's difference.

- HPLC columns need more careful handling and storage and must be flushed with methanol prior to use.
- No such precaution are required for GC columns.

### (iv) Detector difference.

- No such universal HPLC + GC detector has. detector has yet been developed to fulfill these properties of ideal GC detector.
- GC detector has good and reproducible response to solutes, a wide range of linear response, sensitivity and thermal stability.

### (v) Advantages of HPLC over GC

- It is more sensitive, efficient and requires smaller analysis time.
- Less sensitive, inefficient and requires large analysis time.