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$$\text{Vol.} \quad \text{diph} = 0$$

$\text{HCO}_3^-$  is present as alkalinity.

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## TITRIMETRY

Analysis:-

(a) Quantitative

(b) Qualitative

(c) Gravimetric (iii) Titrimetric

Titrimetric analysis was earlier known as volumetric analysis. But volumetric also includes gases. Hence, to avoid this confusion, a new term 'titrimetric analysis' is used.

# TITRATION - Titration is a qualitative analysis of a given compound by neutralising its fixed equivalents with a known solution.

# Types of titrations:-

(a) Acid-base titration. It is also called neutralisation. It includes two steps:-

(i) Acidimetry & Unknown amount of acid is known using standard alkali.

(ii) Alkalimetry & Unknown amount of base is known using standard acid.

(b) Redox titrations.

(c) Complexometric titrations

(d) Precipitation titrations.

For titration, important terms are:-

(a) Titrant - Standard solution.

(b) Titrand - Unknown solution whose concentration is to be found.

(c) Equivalence point - The point at which number of equivalents of titrant equals to the number of equivalents of titrand.

(d) End point - The point at which indicator

(e) Titration error - The small difference b/w equivalence point and end point.

\* Equivalence point is also known as stoichiometric end point or theoretical end-point.

# Standard solution is of two types.

(a)  $1^\circ$  standard

(b)  $2^\circ$  standard.

↳ For a standard to be  $1^\circ$ .

(i) It should be pure.

(ii) Molecular wt. should be accurate to avoid weighing error.

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(iii) It should be highly stable at high temperature ranges.

(iv) It should neither be hygroscopic nor efflorescent  
e.g.,  $K_2Cr_2O_7$ ,  $FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$  (Mohr salt), oxalic acid,  $Na_2CO_3$ ,  $KH(H_4O_4)$  (potassium hydrogen halate),  $KH(IO_3)_2$  (potassium hydrogen iodate).

Examples of  $2^\circ$  titration standard are in  $KMnO_4$ , as  $MnO_2$  is present as impurity in it.  $HCl$ ,  $H_2SO_4$ ,  $NaOH$  and all inorganic acids and alkalies are also  $2^\circ$  standards.

### # Requirements for titration -

(i) It should involve a simple chemical rxn.

(ii) The rxn. should be very fast preferably ionic otherwise catalyst is required.

(iii) Some proper indicators should be available.

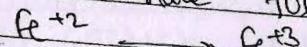
# Indicators - The indicators are basically those compounds used in titration to identify the eq. point. These are of three types.

(a) Internal - Such an indicator is added in the beaker, e.g., starch, methyl orange, phenolphthalein.

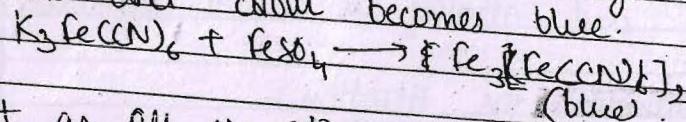
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- (b) External - Used outside the apparatus.
- (c) Self indicator: Either titrant or titrand acts as an indicator, e.g., KMnO<sub>4</sub>

Suppose we have following rxn.

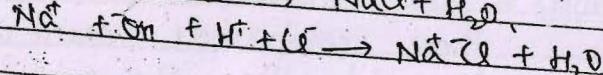
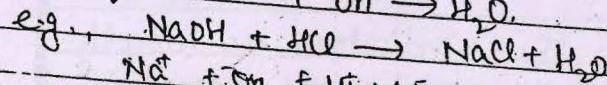
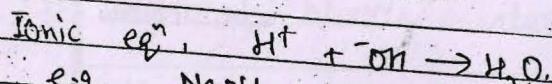


Now to find end-point we introduce K<sub>3</sub>Fe(CN)<sub>6</sub> as an external indicator. We put a drop of K<sub>3</sub>Fe(CN)<sub>6</sub> on a tile and add Fe<sup>+2</sup> drop-by-drop. As far Fe<sup>+2</sup> are present, following rxn. occurs and colour becomes blue.



But as all the Fe<sup>+2</sup> are converted to Fe<sup>+3</sup>, the colour doesn't remain blue.

### ACID-BASE TITRATION:



In acidimetry, base is in burette, [H<sup>+</sup>]<sub>b</sub> and pH increases.

In alkalimetry, acid is in burette [H<sup>+</sup>]<sub>a</sub> and pH decreases.

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Acid-base indicators are complex organic compounds which have different colours in acid & base media. They don't react chemically with acids or bases. Their action can be explained by following two theories - (a) Ostwald's (b) Modern Quinoid's

- (a) Ostwald's Theory:
- (i) They are either weak acids or weak bases
  - (ii) They have different colours in ionised and unionised form.

→ Action of HPh acc. to Ostwald's theory: It is weakly acidic and represented as HPh.

$$\text{HPh} \rightleftharpoons \text{H}^+ + \text{Ph}^-$$

colorless (acidic) Pink, (alkaline)

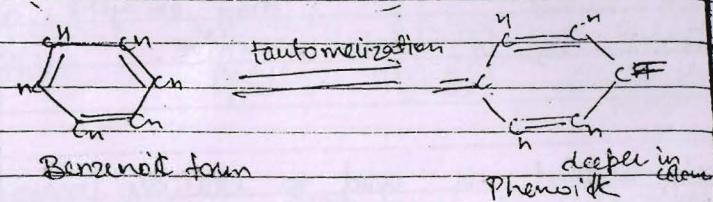
My. action of methyl orange. It is weakly basic and represented as Me

$$\text{Me} + \text{H}_2\text{O} \rightarrow \text{MeH}^+ + \text{OH}^-$$

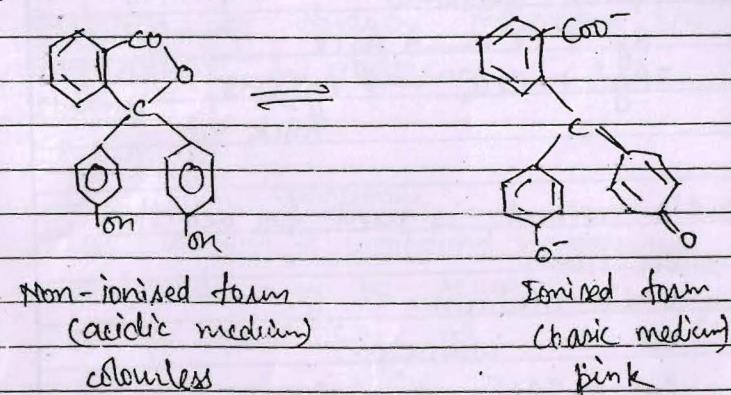
yellow (acidic) red. (basic)

- (b) Modern Quinoid's Theory:
- (i) Acid-base indicators contain

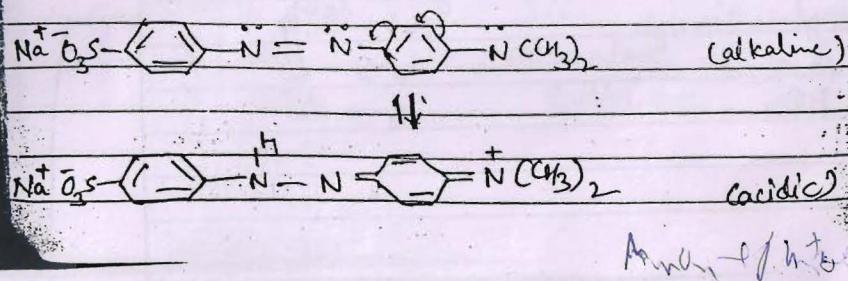
in two forms, either benzoid form or phenoic form.



Action of HPh



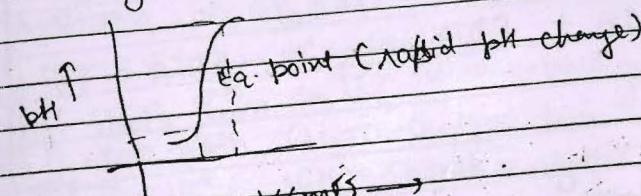
Action of Me:



Acid-base titrations can be of following types.

- Strong acid vs weak base
- Strong acid vs strong base
- Weak acid vs weak base
- Weak acid vs strong base

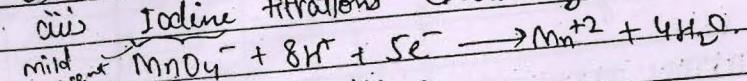
Generally curves are of this type.



(B) REDOX REACTIONS: following two are very ox. agent most important.

i) KMnO4 titrations (with K2Cr2O7 titrations)

ii) Iodine titrations (Colorimetry)

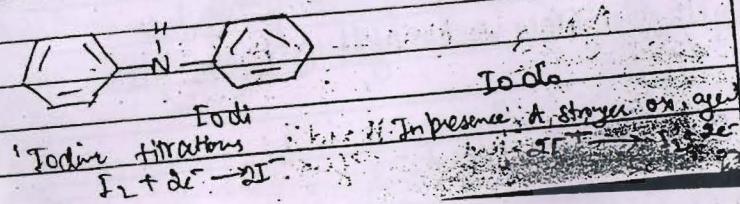


Limitations of KMnO4

a) HCl & HNO3 can't be used.

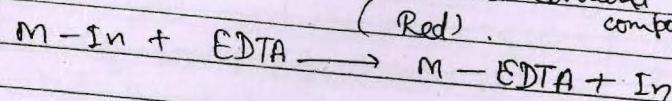
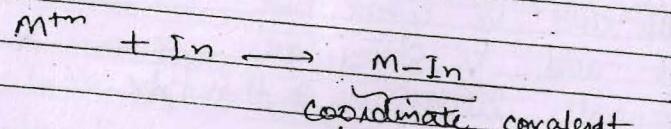
b) It is a 2° standard.

↳ Diphenylamine is commonly used as an internal indicator in redox titrations.



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(C) COMPLEXOMETRIC TITRATIONS = Titrations in which complex is formed

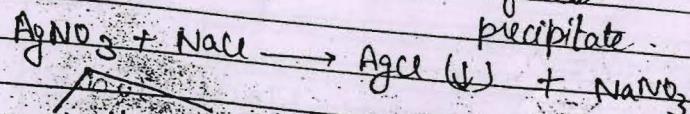


EDTA (Ethylene diamine tetra acetate) is hexadentate. is generally standard solution taken in burette.

Generally EDTA is non-soluble hence its disodium salt is used which has higher solubility.

(D) PRECIPITATION TITRATION:- These are

formation of sparingly soluble salt. It mainly involves  $AgNO_3$ . Hence they are also known as argentometric titration.

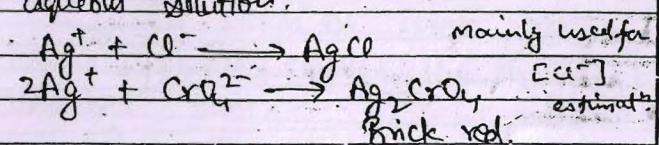


reagent  
Volhard's reagent

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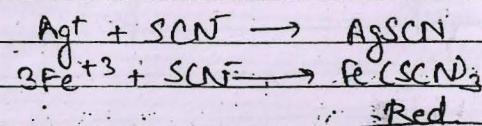
Iodometry  
Iodine cannot be titrated using strong bases  
 $I_2 + NaOH \rightarrow NaI + NaIO_3 + H_2O$   
strong ox. agent  
Moreover  $I_2$  is temp. sensitive.

↳ Mohr's method is used to estimate conc. of halide ions using  $K_2CrO_4$  as indicator in neutral aqueous solution.



↳ Volhard's method is used for detection of silver ions.

Dil. acidic medium  
 $Fe^{+3}$  as an indicator.



SPECTROSCOPY :-

pH ranges of commonly used indicators

Methyl Orange

3.1 - 4.4

M. Red

4.2 - 6

HPh

8 - 10

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React <sup>n</sup>	Titrant	Titrand	Indicator
Neutraliz <sup>n</sup>	Acid/base	base	Hph/M.O.
complexon.	EDTA	M <sup>2+</sup>	
Redox	R.A./O.A.	O.A./R.A.	EBT
Ppt.	AgNO <sub>3</sub>	Ag <sup>+</sup> Cl <sup>-</sup>	Iron chrome black-T K <sub>3</sub> Fe(CN) <sub>6</sub>
			No specific indicator

↳ Spectroscopy is that branch of science which deals with study of structure of compound by studying interactions b/w em. radiations & matter.

\* Advantages:

- Less time consuming
- less amount of compound is required.
- Compound may be reused.
- Highly authentic.

Electromagnetic spectrum is the pattern of arrangement of e.m. waves in order of their wavelength in decreasing freq.

Cosmic rays.

$\gamma$  rays

X rays.

U.V.

Visible

I.R.

Microwave

Radio waves.

U.V. wavelength = 200 - 400 nm

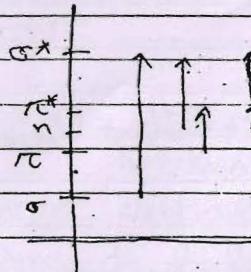
Visible range = 400 - 800 nm  
(Electronic Spectroscopy).

\* UV-visible spectroscopy is mainly used to find conjugation present in a substance.

Transitions possible are

- $\sigma \rightarrow \sigma^*$   $\text{CH}_3, \text{CH}_2, \text{CH}_3$
- $\pi \rightarrow \pi^*$   $\text{CH}_2=\text{CH}-\text{CH}_2$  is non-bonding or lone-pair of e<sup>-</sup>s.
- $\pi \rightarrow \pi^*$   $\text{CH}_2=\text{CH}-\text{CH}_2=\text{CH}-\text{CH}_2$
- $\pi \rightarrow \pi^*$   $\text{CH}_2=\text{CH}-\text{CH}_2=\text{CH}-\text{CH}_2$

Most Highest amount of energy is required in  $\sigma \rightarrow \sigma^*$  as  $\sigma$  e<sup>-</sup>s are highly stable.



Band-diagram

$$E_1 > E_2 > E_3 > E_4$$

\* Absorbance is additive property

Transmittance is multiplicative property.

v<sub>2</sub>

$$\frac{mv}{\sqrt{1-v^2/c^2}}$$

m<sub>0</sub><sup>2</sup>/v<sup>2</sup>

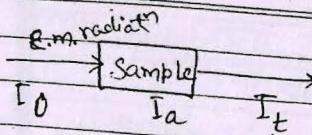
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$$\text{Transmittance}, T = \frac{I_t}{I_0} = \frac{\text{Intensity of transm. light}}{\text{Intensity of incident light}}$$

$$\begin{aligned}\text{Absorbance} &= -\log T \\ &= -\log \left( \frac{I_t}{I_0} \right).\end{aligned}$$

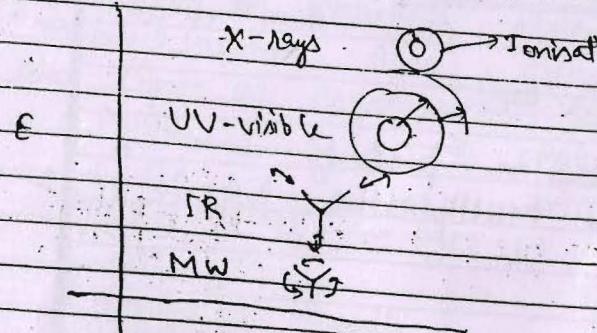
$$A = \log \left( \frac{I_0}{I_t} \right)$$

- Spectra are of two types:
- Atomic/electronic spectra
  - Molecular spectra

$$E_T = E_{\text{translat}} + E_{\text{vibrat}} + E_{\text{rotat}} + E_{\text{electrom}}$$

$$E_T < E_n < E_v < E_{ee}$$

X-rays  $\rightarrow$  Ionisation



Lambert's Law: When a monochromatic light is passed through a sample soln., the decrease in intensity of light with the thickness of solutn. is directly proportional to intensity of incident light.

$$-\frac{dI}{dx} \propto I$$

$$-\frac{dI}{dx} = kI$$

$$-dI = k dx$$

$$I = I_0 \text{ at } x=0$$

$$I = I_t \text{ at } x=l$$

$$-\left[ \log I \right]_{I_0}^{I_t} = k(l)_0^l$$

$$\Rightarrow -\log \left( \frac{I_t}{I_0} \right) = kl$$

$$\alpha : \frac{I_t}{I_0} = e^{-kl}$$

$$\alpha : 2.303 \log \frac{I_t}{I_0} = -kl$$

$$\log \frac{I_t}{I_0} = -\frac{kl}{2.303}$$

$$\alpha : A = \epsilon l$$

$\epsilon$  = Molar extinction coefficient  
or molar absorptivity

Bear's law Rate of change of intensity w.r.t. change of length thickness of atm. is directly proportional to intensity of atm. as well as its concentration.

$$-\frac{dI}{dx} \propto I$$

$$-\frac{dI}{dn} \propto c$$

$$\therefore -\frac{dI}{dx} \propto Ic$$

$$\therefore -\frac{dI}{dx} = kI dn$$

On integrating & solving,  
we get,

$$A = \epsilon cl$$

This is called Bear's atm. or Lambert- Bear solution.

Q. At 460 nm a blue filter transmits 72.7% & a yellow filter 40.7%. What is the transmittance of both in combination atm?

$$T_1 = \frac{72.7}{100}$$

$$T_{\text{Total}} = T_1 \times \frac{40.7}{100}$$

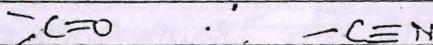
$$\Rightarrow 29.58\%$$

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### Instrumentation

Chromophores : literal meaning - colour carrier. Initially any group which was responsible for imparting colour, was considered as a chromophore. But now this term has been replaced for any covalently bound unsaturated group which absorbs in UV region without considering the fact it produces colour or not, e.g.,



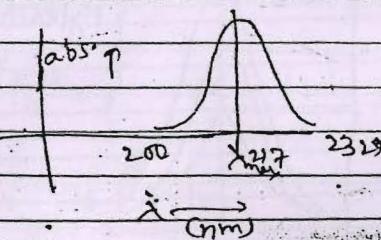
Chromophores can be of two groups.

- (i)  $\pi \rightarrow \pi^*$  transition is taking place.
- (ii)  $n \rightarrow \pi^*$  transitions are taking place.
- $\pi \rightarrow \pi^*$

Auxochromes : literal meaning - colour enhancers.

Auxochromes are those grp. which when present along-with a chromophore, shift  $\lambda_{\text{max}}$  to a longer wavelength & cause bathochromic shift (red).

$\lambda$  at which absorption is maximum is called  $\lambda_{\text{max}}$ .

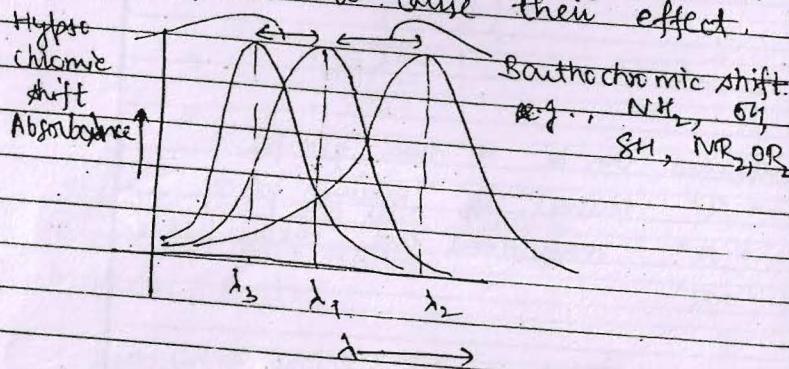


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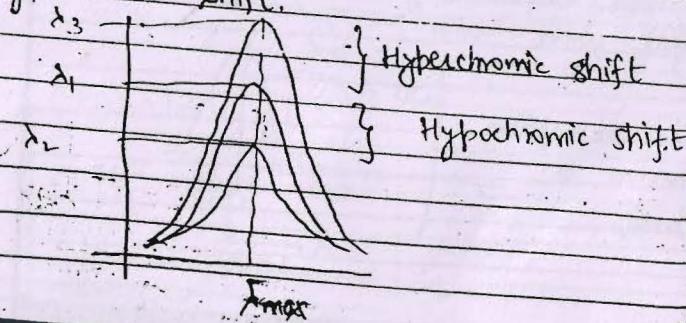
e.g., In  $\text{CH}_2=\text{CH}-\text{CH}=\text{CH}_2$ , if  $\lambda_{\text{max}}$  is 217 nm.

but in presence of -OH as auxochromes in  $\text{CH}=\text{CH}-\text{CH}=\text{CH}-\text{OH}$ ,  $\lambda_{\text{max}}$  is 232 nm.

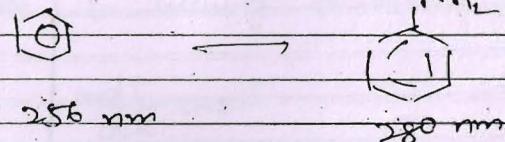
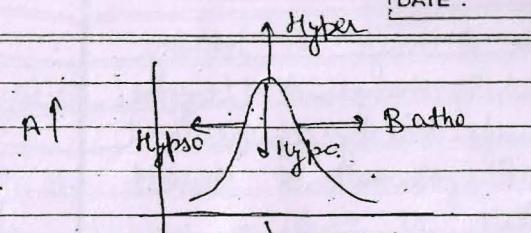
- Auxochromes don't affect absorption but only cause 'Baethochromic shift' & They should be in conjugation with chromophores to cause their effect.



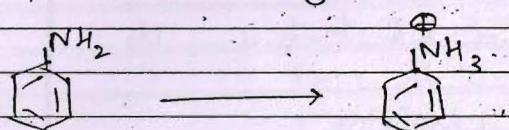
\* Intensity of absorption increases only and there is no change in  $\lambda_{\text{max}}$  is called hyperchromic shift.



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$-\text{NH}_2$  extends conjugation & causes red shift



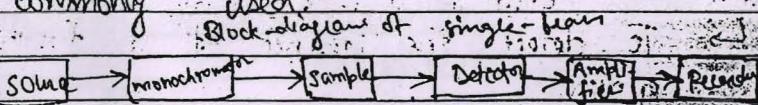
Hypoehromic shift due to removal of conjugation

SPECTROPHOTOMETER :- Describes the amount of light absorbed & transmitted when UV-visible light is produced

These are of two types:-

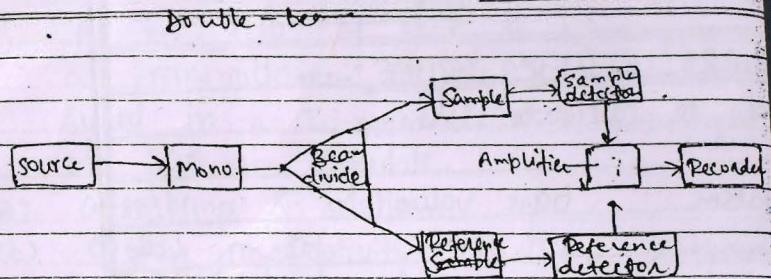
(a) Single beam      (b) Double beam

Nowadays double beam are commonly used.



UV range 200 - 800 nm

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In UV Spectroscopy Hg / D<sub>2</sub> discharge lamp & invisible tungsten lamp is used.

Beam splitter divides monochromatic light in two equal parts.

Monochromatir is generally a prism.

Sample detector detects spectra of (sample + solvent) & reference detector detects spectrum of solvent only.

Amplifier acts as an electronic balance. Recorder records graph of  $\text{A}_{\text{max}}$ .

## # APPLICATIONS :-

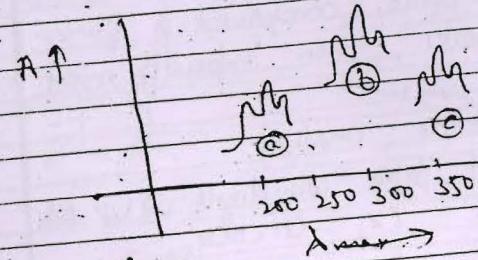
① Usually used to study the extent of conjugation present in molecule.

Extent of conj.  $\propto \lambda_{\text{max}}$

② To detect the presence of a functional group.

To determine the presence of conjugation.

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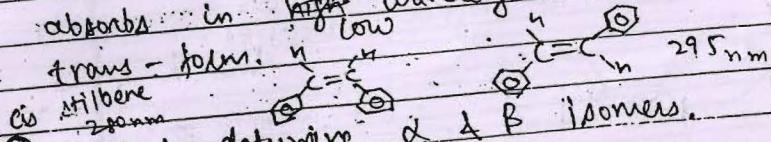
Find a, b, c,

- (i)
- (ii)
- (iii)

$$a = 2, b = 1, c = 3.$$

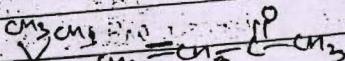
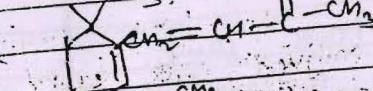
③ Configurations of Phenols & aromatic amines (ext of conjugation)

④ Used to determine geometrical isomers. cis-isomers are more strained i.e. high amount of energy is req. Hence it absorbs in low wavelength than its trans-form.



⑤ Used to determine A & B isomers.

$$a = 280 \text{ nm}, b = 236 \text{ nm}$$



⑥ To study chemical kinetics.

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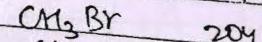
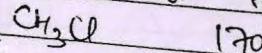
↳ ⑦ Used to find purity.  
If one sharp peak, compound is pure.

If several peaks, then impurity might be present. Using Lamb's law

↳ ⑧ Qualitative & Quantitative analysis

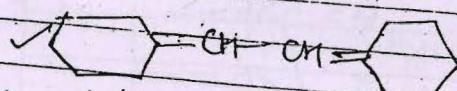
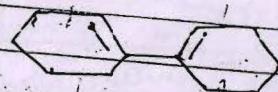
Q. Which will exhibit higher wavelength in UV-vis?  
a)  $\text{CH}_3\text{COCH}_3$  or  $\text{CH}_2=\text{CHCOCH}_3$ .  
Ans: second one.

Q. Explain reason for difference in absorption.



Ans. As outer nuclear interact ↓ due to ↑ in size, less energy is reqd. Hence higher  $\lambda_{\text{max}}$ .

Q. Which absorbs at higher wavelength?



Ans. As both seem to have same extent of conjugation, we cannot decide. We use Woodward's Fisser's rule.  
1 has higher wavelength.

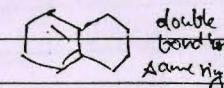
### # WOODWARD FISHER'S RULE :-

Used to calculate  $\lambda_{\text{max}}$

System base value

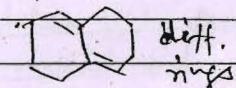
Homoannular

253 nm



Heteroannular

217 nm



#### increments

Exocyclic double bond = 5

Ring residues = 5

Alkyl substituents = 5

-X = 5 five.

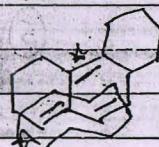
-OH = 6

-OR = 6

-NH = 30

-NR = 60

desirable bond extending conjugation = 30



Two exocyclic double bonds.

base part:

line attached with basic system constitute ring residues.

These are part of ring also & conj. system also.

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ACD - T

Q. A  $7.25 \times 10^{-5}$  M soln. of KMnO<sub>4</sub> has a transmittance of 44.1% when measured in a 2.1 m cell at a wavelength of 525 nm. Calculate.

(a) absorbance of solution.

(b) molar absorptivity (extinction) coefficient of KMnO<sub>4</sub> solution.

$$\text{Absorbance} = -\log T \\ = -\log \left( \frac{44.1}{100} \right) \\ = 2.164$$

Also  $A = \epsilon cl$ .

$$\epsilon = \frac{A}{cl} = \frac{2.164}{7.25 \times 10^{-5} \times 2.1 \times 10^{-2}} \\ = 3.6 \times 10^5$$

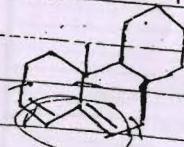
$= 2.36 \times 10^5$  approx

Q. Find  $\lambda_{max}$



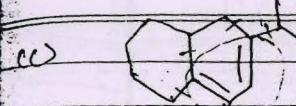
d.l.m.(a)  $253 + 5 \times 2 = 263 \text{ nm}$

(b)



d.l.m.(b)  $217 + 3 \times 3 = 237 \text{ nm}$

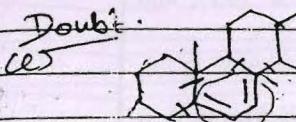
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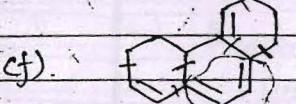
d.l.m.(c)  $253 + 5 \times 4 + 5 = 278 \text{ nm}$



d.l.m.(d)  $217 + 5 \times 4 + 5 = 242 \text{ nm}$



d.l.m.(e)  $253 + 5 \times 4 = 273 \text{ nm}$



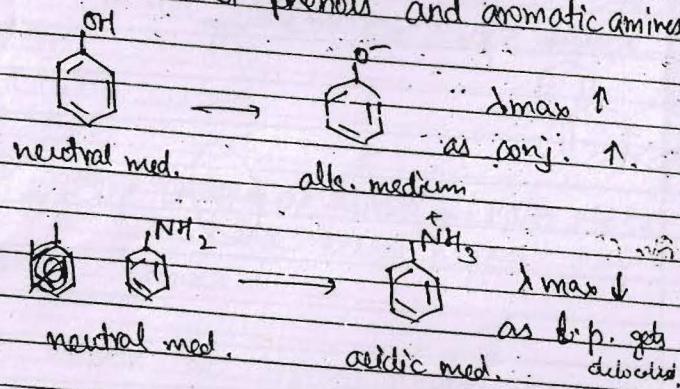
d.l.m.(f)  $253 + 5 \times 4 + 30 + 30 + 5 = 338 \text{ nm}$

1  
2  
3

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Uses of spectroscopy:

1. Chromophore.
2. Extent of conjugation
3. Differentiate b/w conj. & nonconj. systems.
4. " geometrical isomers.
5. Qualitative analysis.
6. Quantitative analysis using Lambert's Beer Rule.
7. Chemical kinetics.
8. Detection of impurities.
9. Confirmations of phenols and aromatic amines.



### WOODWARD'S FRIESER'S RULE I

1. Acyclic / Heterocyclic
2. Homoannular
3. Acyclic / cyclic 6 membered  $\alpha, \beta$ -unsaturated ketone/ ald.  $\lambda_{\max} = 215$
4. 5-membered unsaturated ketone  $\lambda_{\max} = 202$

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5. 5-membered unsaturated aldehyde 207.

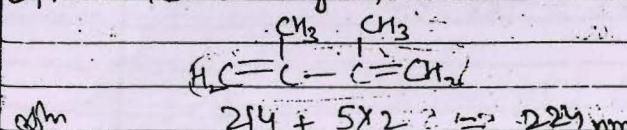
Increments:

Alkyl substituent	5
Ring residues	5
Exocyclic double bond	5
-OH, -X	6
SR	30
NR <sub>2</sub>	60
double bond extended conj.	30.

For  $\alpha, \beta, \gamma, \delta$  unsaturated aldehydes / ketones increments are:

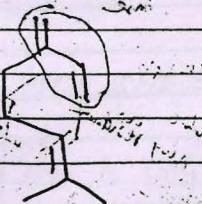
$\alpha$	10
$\beta$	12
$\gamma$	18
$\delta$	18
H. Annular	39
Heteroannular	32

Q. 2,3 dimethyl 1,3 butadiene. Find  $\lambda_{\max}$



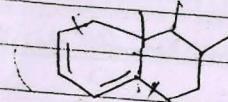
Q. Find  $\lambda_{\max}$

$$\text{CH}_3 \quad 214 + 5 = 219$$

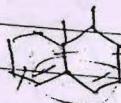


Q. Find  $\lambda_{max}$

$$\text{dm. } 253 + 5 + 5 \times 3 \\ = 253 + 20 \\ = 273 \text{ nm.}$$

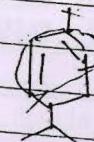


Q. Find  $\lambda_{max}$



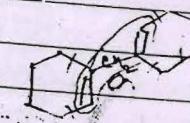
$$\text{dm. } 214 + 15 + 5 \\ \text{exo.} \quad 284 \text{ nm.}$$

Q. Find  $\lambda_{max}$

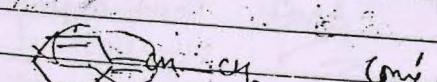


$$\text{dm. } 253 + 5 \times 3 = 268.$$

Q. Find  $\lambda_{max}$ .



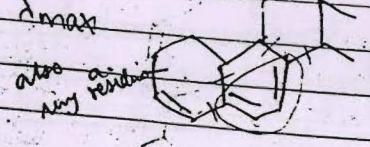
no conj.



conj.

$$\text{dm. } 2. \lambda_{max} 214 + 5 + 5 \times 3 = 284 \\ \text{base only my res.}$$

Q. Find  $\lambda_{max}$



in ethanol.

also acidic  
my res.

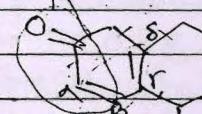
$$\text{dm. } 253 + 30 + 5 + 20 + 5 + 5$$

$$= 258 + 50 + 5 = 308 + 5 \\ = 313 + 5$$

basic

$$= 318$$

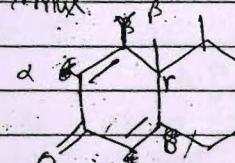
Q.



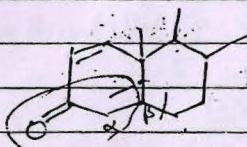
$$\text{dm. } 215 + \cancel{39} + 20$$

$$= 215 + 39 + 30 + 18 + 36 = 338. \\ \text{d. b. extended} \\ \text{H-Aromatic conj.}$$

Q. Find  $\lambda_{max}$



$$\text{dm. } 215 + 18 + 36 = 215 + 36 + 18 \\ = 215 +$$



base system as it is highly substituted  
exocyclic d.b.

$$= 215 + 5 + 24 = 244.$$

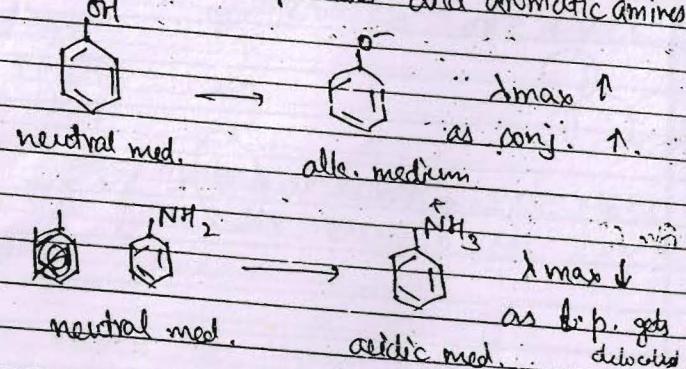
Q. find  $\lambda_{max}$

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Uses of spectroscopy:

1. Chromophore.
2. Extent of conjugation.
3. Differentiate b/w conj. & unconj. systems.
4. " geometrical isomers.
5. Qualitative analysis.
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7. Chemical kinetics.
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9. Confirmations of phenols and aromatic amines.



### WOODWARD'S & FIESER'S RULE I

1. Acyclic / Heterocyclic
2. Homoannular
3. Acyclic / cyclic 6 membered  $\alpha, \beta$  unsaturated ketone/ ald.
4. 5-membered unsaturated ketone 202

Base value (nm)

214

213 in some books.

253

### 5-membered unsaturated aldehyde 207

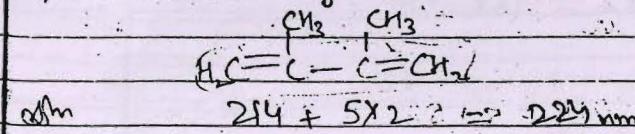
#### Increments:

Alkyl substituent	5
Ring residues	5
Exocyclic double bond	5
-OH, -X	6
SR	30
NR <sub>2</sub>	60
double bond extended conj.	30

For  $\alpha, \beta, \gamma, \delta$  unsaturated aldehydes / ketones increments are:

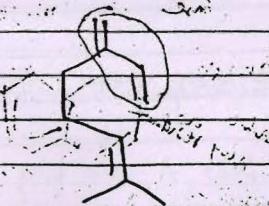
$\alpha$	10
$\beta$	12
$\gamma$	18
$\delta$	18
H. Aromatic	39
Heteroaromatic	32

Q.  $\alpha, \beta$  dimethyl 1,3 butadiene. Find  $\lambda_{\max}$



Q. Find  $\lambda_{\max}$

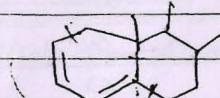
$$\Delta m: 214 + 5 = 219$$



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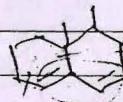
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Q. Find  $\lambda_{\text{max}}$



$$\begin{aligned} \text{dm. } & 253 + 5 + 5 \times 3 \\ & = 253 + 20 \\ & = 273 \text{ nm.} \end{aligned}$$

Q. Find  $\lambda_{\text{max}}$



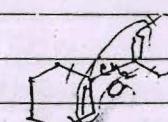
$$\text{dm. } 214 + 15 + 5 = 234 \text{ nm.}$$

Q. Find  $\lambda_{\text{max}}$



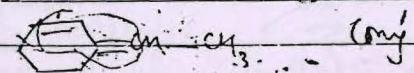
$$\text{dm. } 253 + 5 \times 3 = 268.$$

Q. Find  $\lambda_{\text{max}}$ .



no conj.

a

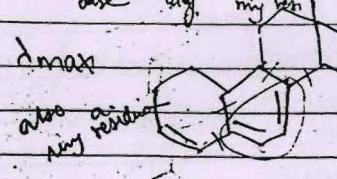


conj.

$$\text{dm. } 214 + 5 + 5 \times 3 = 284$$

base soln. my mi

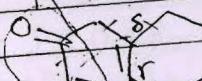
Q. Find  $\lambda_{\text{max}}$



in ethanol

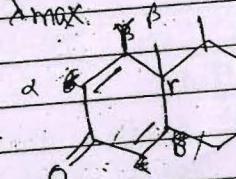
$$\begin{aligned} \text{dm. } & 253 + 30 + 5 + 20 + 5 + 5 \\ & = 258 + 50 + 5 = 308 + 5 \\ & = 313 + 5 \\ & = 318 \end{aligned}$$

basic

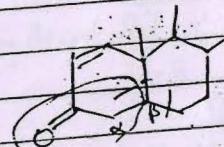


$$\begin{aligned} \text{dm. } & 215 + \cancel{39} + 20 + \cancel{39} + 18 + 36 = 338. \\ & = 215 + 39 + 30 + 18 + 36 \text{ (r. ir. S)} \\ & \quad \text{d. extended} \\ & \quad \text{H-Aromatic conj.} \end{aligned}$$

Q. Find  $\lambda_{\text{max}}$



$$\begin{aligned} \text{dm. } & 215 + 18 + 36 = 215 + 36 + 18 \\ & = 215 + \end{aligned}$$



base system as it is highly substituted

exocyclic d. b.  $\beta$ -acidic

$$- 215 + 5 + 29 = 244.$$

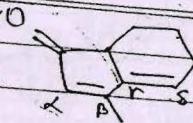
Q. Find  $\lambda_{\text{max}}$

soln.

2  
3

only show those molecules will be IR active which have a change in net dipole moment.

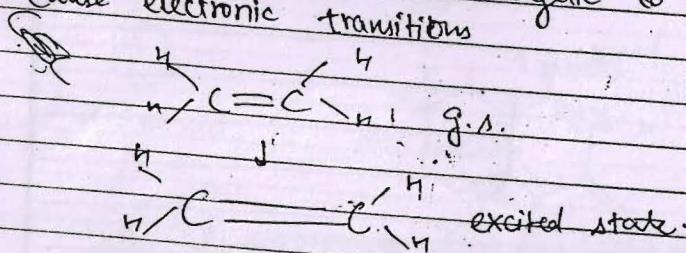
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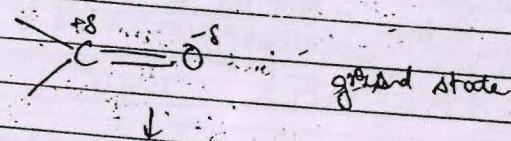
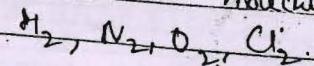
$$\text{M.M.} \quad 20.9 + 12 + 18 + 18 + 30 + 5 \\ = 232 + 48 + 5 \quad 280 + 5 = 285 \\ 4000 - 650 \text{ cm}^{-1}$$

# IR SPECTROSCOPY → Cause only vibrations.

These diff. vibrations cause atoms to vibrate. They are not that much energetic to cause electronic transitions.



No change in dipole moment. ∴ I.R. inactive.  
e.g., homodiatomic molecules



Dipole moment change. ∴ I.R. active

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Molecular vibrations can be classified as under:

### Vibrations

B.A. position  
unchanged

stretching

R.A. position  
changes

bending

Symm. Asymm.

In plane Out of plane

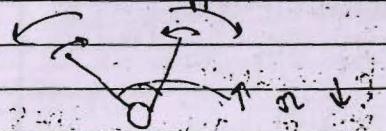
wagging

scissoring Rocking

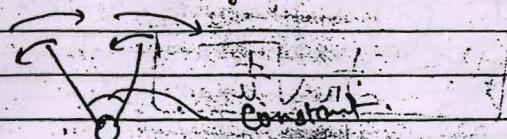
Twisting

Positn of all the  
bond-axis with respect to  
remains unchanged. current atom

Scissoring: Either both come together to go opposite. Bond angle either ↑ or ↓.



Rocking: Bond angle remains constant



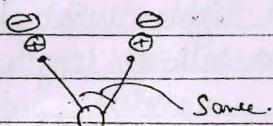
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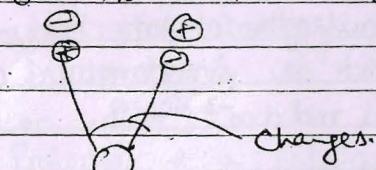
225

24

Wagging :- Both bonds either go into the plane or come outside of plane.



Twisting :- One inside other outside or vice-versa



↳ For non-linear polyatomic molecules.

Total no. of fundamental vibrations =  $3n - 6$

Out of these where  $n$  is no. of atoms in molecule.

No. of stretching vib. =  $(n-1)$

No. of bending vib. =  $2n-5$

↳ For linear p. molecules.

Total no. of f.v. =  $3n-5$

Stretching =  $n-1$

Bending =  $2n-4$

$$V = \frac{1}{2\pi} \sqrt{\frac{k}{\mu}}$$

$f$  is force constant.

for single bond  $f = 5 \times 10^3$  dyne/cm.

for double "  $f = 2 \times 5 \times 10^5$  ..

for triple "  $f = 3 \times 5 \times 10^5$  ..

$\mu$  = reduced mass =  $\frac{m_1 m_2}{m_1 + m_2}$

Q. Out of following pairs which one is expected to absorb at higher frequency for stretching vibrations.

(a)  $C=C$  or  $C-C$ .

(b)  $C=C$ .

(c)  $O-H$  or  $C-C$ .

(d)  $C-C$  as  $\mu$  will be lower ~~?~~

Q. Calculate approx. freq. of C-H stretching

$$k = 500 \text{ N m}^{-1}$$

$$\text{mass of } C = 20 \times 10^{-24} \text{ g.}$$

$$\text{mass of } H = 1.6 \times 10^{-24} \text{ g.}$$

$$\therefore \mu = \frac{20 \times 1.6 \times 10^{-24}}{21.6} \text{ g.}$$

$$f = \frac{1}{2\pi} \sqrt{\frac{2 \times 500 \times 21.6}{21.6 \times 16}} \times 10^{-12}$$

$$= \frac{1}{2\pi} \times \frac{5}{4} \times \frac{8 \times 56}{21.6} \times 10^{-12} \text{ Hz.}$$

Note :- T.R. activity also depends on type of vibrations.

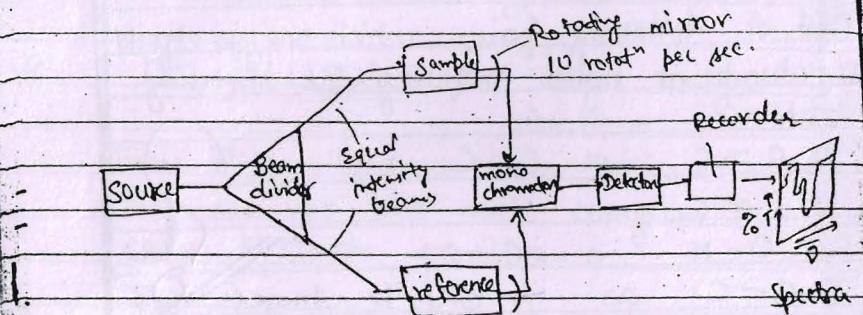
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### Instrumentation:

Source is electrically heated Nernst filament  
(consists of oxides of Zr, cerium & thorium)  
or globar red (red at silicon carbide)

Temperature reached is  $1000^{\circ}\text{C}$ .



Sample & reference cells cannot be made of glass or quartz as they absorb in IR range & results in wrong results.

Alkali halides are used. KBr is used in case of solids in form of pellets.

for liquids, we use IR inactive solvents e.g., Nujol (a transparent hydrocarbon) as KBr can't be used.

Chloroform, ccl<sub>4</sub> can also be used.

### Practice Problems

Q.1. A certain substance in a cell of length 1 absorbs 10% of incident light. What fraction will be absorbed in a cell of length 5 times as long?

Q.2. In a given absorption cell, transmittance of 0.1 mol dm<sup>-3</sup> is 0.75 and 0.1 mol dm<sup>-3</sup> B is 0.55 at a given wavelength. Calculate transmittance of a soln. which is simultaneous 0.1 mol dm<sup>-3</sup> in A and 0.1 mol dm<sup>-3</sup> in B.  $\rightarrow 0.98$

$$\text{Soln. 2. } T = T_1 \times T_2 \\ = 0.55 \times 0.75 \\ = 0.4125.$$

$\log_{10} \frac{T}{T_0} = -\epsilon cl$   
 $\therefore \epsilon = \frac{\log_{10} \frac{T}{T_0}}{cl}$

$$\text{Soln. 1. } A = \Sigma c_i l_i \\ = \Sigma \frac{\text{no. of mol.} \times l_i}{Molar mass} \times \epsilon_i l_i \\ = \frac{\text{no. of mol.} \times l_i}{Molar mass} \times \epsilon_i l_i$$

$$I_a = 10\% \text{ of } I_0 \quad \therefore I_t = 90\% \text{ of } I_0$$

$$A = \log \left( \frac{I_0}{I_t} \right) = \log \left( \frac{10}{9} \right)$$

Also  $A = \log \left( \frac{I_0}{I_t} \right)$

$$\therefore A = A_{10} = \log (0.9)$$

$$\therefore I_{10} = I_0 \times (0.9)^{10} = 39.37\% \text{ of } I_0$$

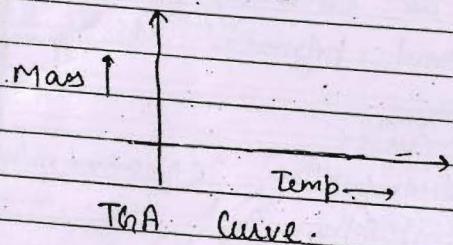
$$\therefore I_{10} = 50.625 \times 40.98 \text{ of } I_0$$

## Thermal methods of Analysis

It is that part of science in which a substance is analysed by studying its changes w.r.t. temperature.

- (in symbol)
- (i) TGA
  - (ii) Thermo Gravimetric Analysis
  - (iii) DTA
  - Differential Thermal Analysis
  - (iv) DSC
  - Differential Scanning Calorimetry
  - (v) Dil.
  - Dilatometry. (vol. is used)

TGA = Properties of a substance or reaction products are analysed by studying the mass change w.r.t. temp. change.



### Instruments :-

- (a) Microbalance (Thermobalance). Range 0.1 mg to 100 mg
- (b) Furnace (20°C to 1000°C)  
But some furnaces can produce upto 1600°C.  
They have cooled with forced air. Some can cool upto from 1000°C - 50°C. in 20 min.

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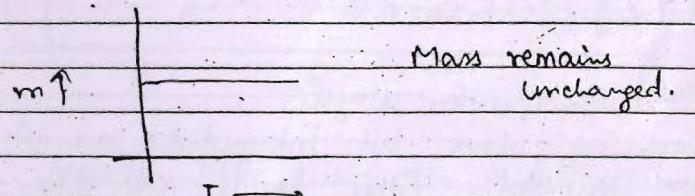
Heating rate is ~~is~~  $0.1^\circ\text{C min}^{-1}$  -  $100^\circ\text{C min}^{-1}$   
In some cases it is upto  $200^\circ\text{C min}^{-1}$ .

(c) Purgung System :- To provide inert atmosphere in the furnace.  
So that error is not introduced.  
 $\text{N}_2$ ,  $\text{He}$ ,  $\text{Ar}$  etc. used.

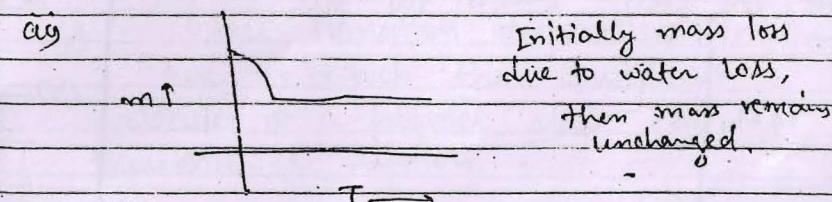
(d) Recorder :- Records the data & gives analysis

### Types of curves.

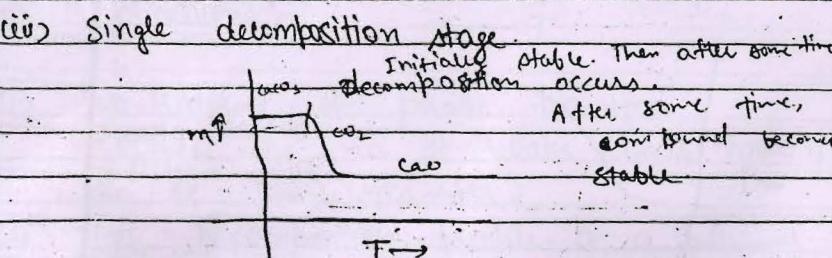
(i) Straight line. Compound is highly stable.



Mass remains unchanged.



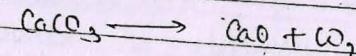
Initially mass loss due to water loss, then mass remains unchanged.



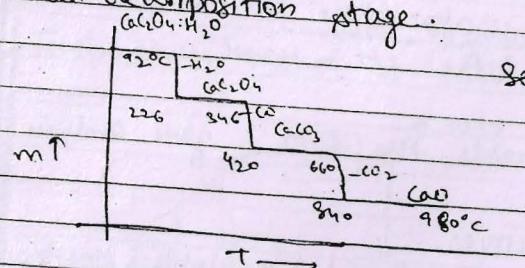
Initially stable. Then after sometime decomposition occurs.  
After some time, compound becomes stable

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e.g.,  $\text{CaCO}_3$



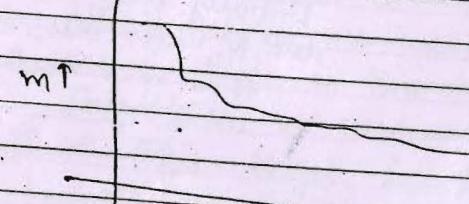
(iv) Multi decomposition stage.



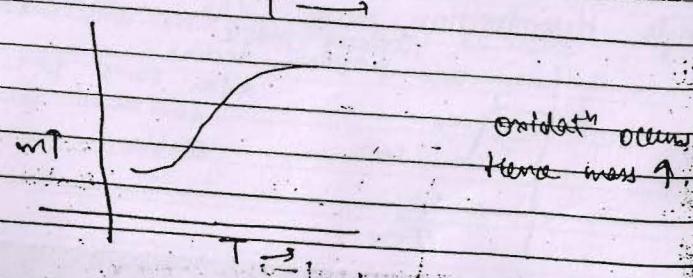
e.g.,  $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$

(v) Multi decomposition stage.

Same as above but intermediates are not separated. Either heating rate is too fast or react<sup>n</sup> rate is too fast.



(vi)



oxidat<sup>n</sup>

m<sup>f</sup>

T →

oxidat<sup>n</sup> occurs  
but intermediate  
is unstable  
& decomposes

### # APPLICATIONS :-

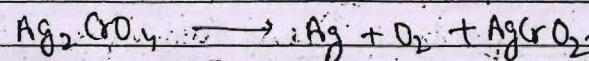
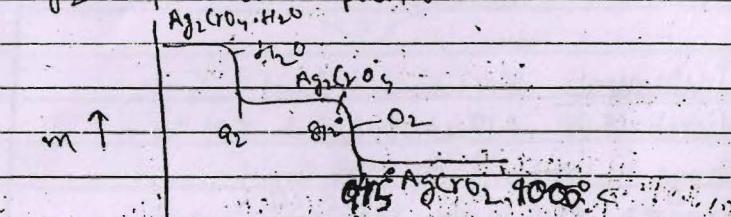
- To determine the thermostability of a compound.
- Composition of compound.
- Decomposition profile.
- Moisture & volatile content of a compound.

TGA curves are extremely useful in pharmaceutical and polymer industry.

↳ Some useful TGA curves

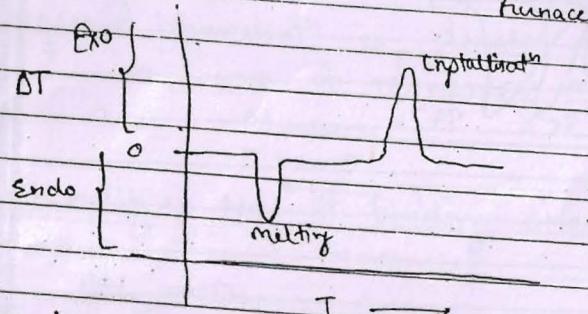
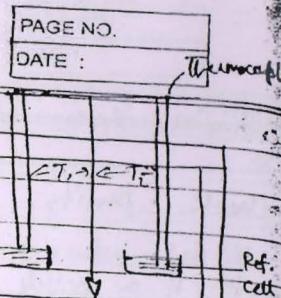
(a)  $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$  decomposition. (Discussed earlier)

(b)  $\text{Ag}_2\text{CrO}_4$  decomposition.

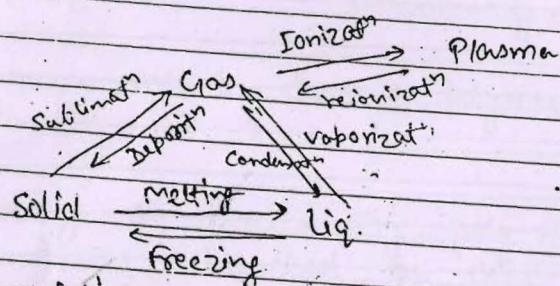


### DTA :

$\Delta T$  is taken into account of both the sample as well as reference cell.



### Phase transition :



### Instrumentation :

(a) Sample holder Thermo couple is created b/w reference and sample cells. A furnace is placed to provide inert atmosphere. A voltmeter detects temp. changes as it is connected with thermo couple.

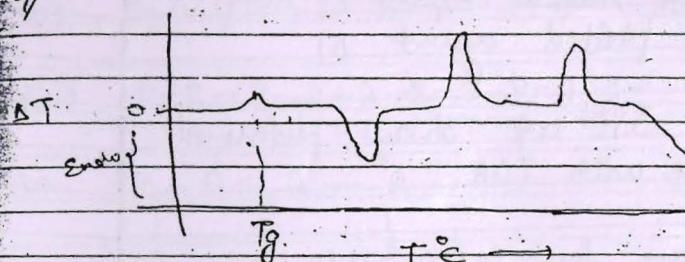
### DTA furnace

(b) Sample container.

(c) Programmer - Runs a constant temperature program software.

### Recorder

### CURVE :



A typical DTA curve for a polymer

T<sub>g</sub> : Glass transition temperature. Temperature at which the constituent molecules of a polymer start executing translational motion.

### Applications :

(i) % Amount of phase transfer (Higher the no. of peaks / lower more no. of phase transfer)

(ii) Decomposition profile of a polymer.

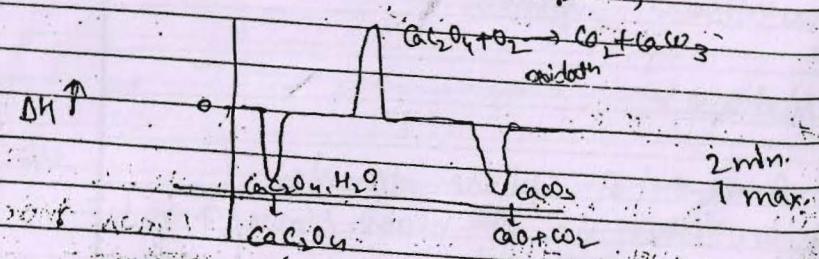
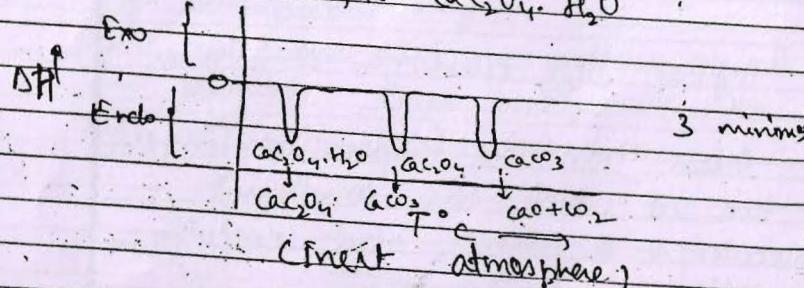
iii) Thermal stability of a polymer.

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# DSC Differential Scanning Calorimetry  
 AH (Enthalpy change) is measured  
 Amount of energy req. to heat both the  
 reference & sample at const. temp.  
 AH is plotted against AT.

DTA & DSC are always performed  
 together with TGA.

DSC curve for  $\text{CaCO}_3 \cdot \text{H}_2\text{O}$



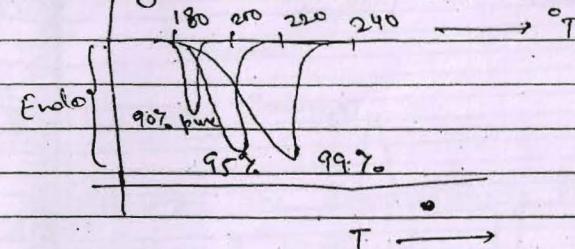
Energy (mJ/min) presence of  $\text{O}_2$

Application:

DSC =

iii) To check purity

on melting of a solid.



# POLYMERS

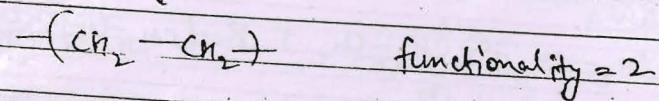
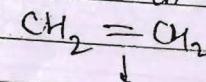
A no. of repeating units combine to form a large chain.

All polymers are macromolecules but all macromolecules are not polymer.

Degree of polymerisation is denoted by "n".

$$M = M_{\text{polymer}} - M_{\text{monomer}}$$

# functionality = No. of binding sites present in a monomer.



for a monomer to form a polymer,  
functionalities  $\geq 2$ .

\* Depending on functionality, polymers can be linear, branched or cross-linked

## # Classification of Polymers:

- (a) On basis of type of monomer  
 (i) Homopolymer — One type of monomer  
 (ii) Copolymer — More than one monomers

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$$nA \rightarrow -[A]_n^- \quad \text{Homopolymer}$$

$$nA + nB \rightarrow -(A-B)_n^- \quad \text{copolymers}$$

↳ On basis of arrangement of monomers, copolymers can be of four types.

iii)  $A - B - A - B - A - B -$  Alternating

wj A-A-A-B-B-A-B- Random.

(iii)  $B-B-B-A-A-B-B-B-A-A-$  Block

A - A - A - A

|      |      |

B      B      B

(b) On basis of structure of polymer  
 cis Linear  $\rightarrow$  higher density due to more closed packing  
 trans Branched

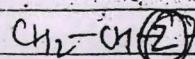
(iv) Cross-linked : highest density; stiffener

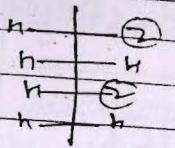
(c) On the basis of stereochemistry or tacticity

(i) Isotactic

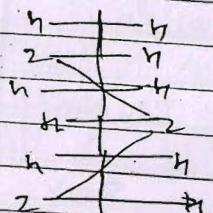
(ii) Syndiotactic arrangement

### (cii) Atacticity:

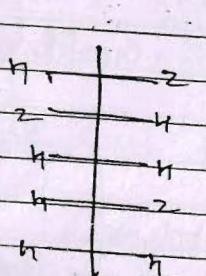




On same side of chain  
∴ Isotactic



Syndiotactic



No proper arrangement  
∴ Atactic

(d) On basis of synthesis.

(i) Addition (Step growth)

Initiat<sup>n</sup>, propagat<sup>n</sup>, terminat<sup>n</sup>

(ii) Condensation (Chain growth)

Addit<sup>n</sup> reactions can be of three types

(a) free radical addition polymerisation

(b) Ionic addition polymerisation

(c) Co-ordination addition polymerisation

## FREE RADICAL ADDITION

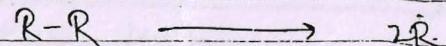
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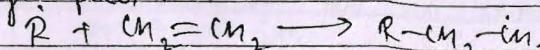
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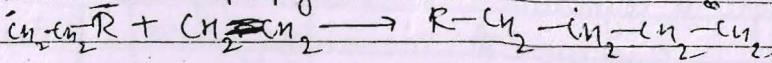
### (i) Chain initiation.



R is usually benzoyl peroxide or tert-butyl peroxide



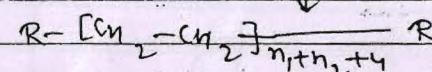
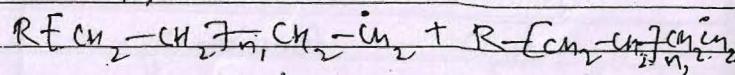
### (ii) Chain propagation.



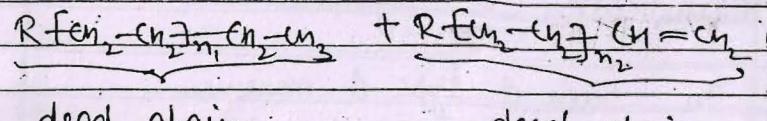
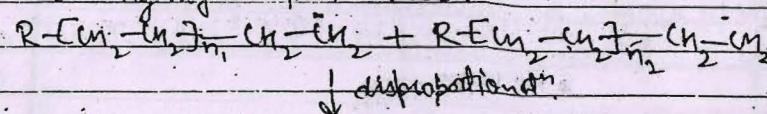
### (iii) Chain Termination

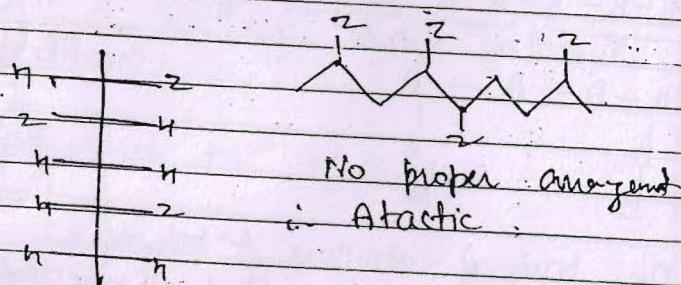
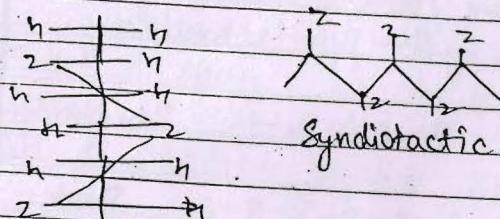
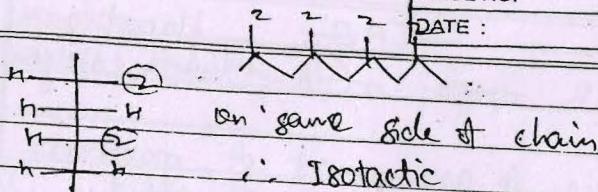
It can be done by either of addit<sup>n</sup> disproportionation or chain transfer

In addit<sup>n</sup>, we add two free-radical chains,



In disproportionat<sup>n</sup>, one chain acquires a hydrogen from other.





(d) On basis of synthesis:

(i) Addition (Step growth)

Initiation, propagation, termination

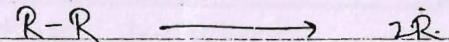
(ii) Condensation (Chain growth)

Addition reactions can be of three types:

- (a) free radical addition polymerisation
- (b) Ionic addition polymerisation
- (c) Co-ordination addition polymerisation.

## FREE RADICAL ADDITION

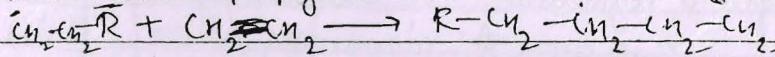
(i) Chain initiation.



R is usually benzoyl peroxide or tert-butyl peroxide



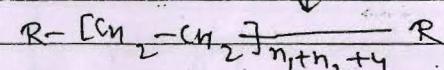
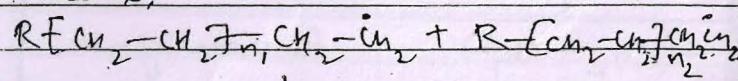
(ii) Chain propagation.



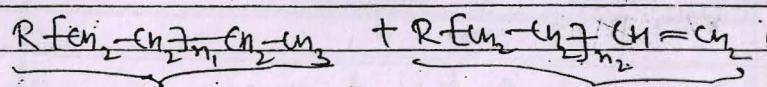
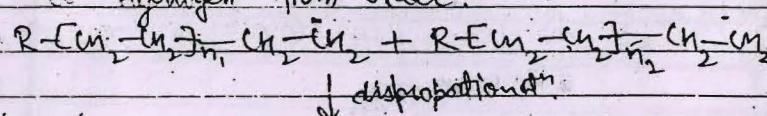
(iii) Chain Termination

It can be done by either of additivity  
disproportionation or chain transfer

In addit<sup>n</sup>, we add two free-radical chains.



In disproportionation, one chain acquires a hydrogen from other.

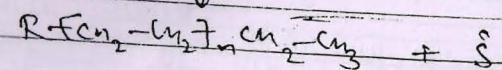
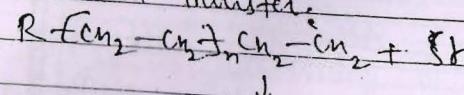


dead chain

dead chain

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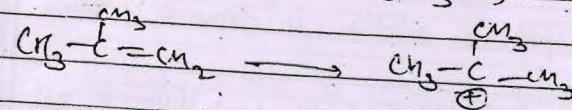
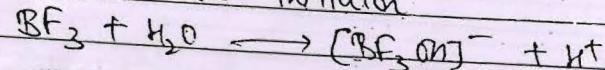
## In chain transfer



## # IONIC POLYMERISATION:

Can be of two types cationic & anionic  
Cationic

Takes place in presence of strong protic acid or Lewis acid and traces of water act as initiator



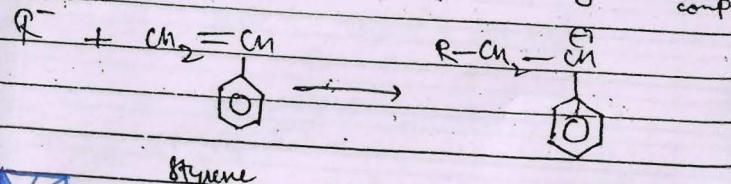
In "feminat", On attacks & she is beaten

### Anionic

Cation is generated from original  
reagent  $\text{R}_2\text{MgX} \rightarrow \text{R}^+ + \text{MgX}_2^-$

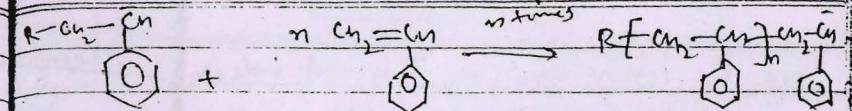
$$R - \text{O}^+ + \text{H}_2\text{O} \rightarrow R - \text{OH}^- + \text{H}_3\text{O}^+$$

An anion is generated from a nonmetal.



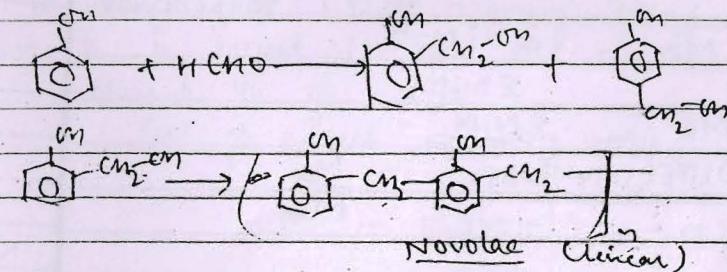
$\text{CH}_2=\text{CH}-\text{CH}_2$

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# CONDEMNATION

## Phenol-formaldehyde resin (Pf resin).

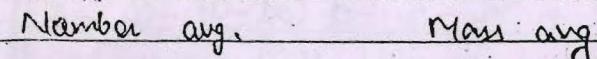


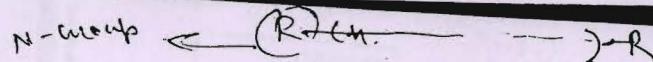
When O & P condense, they give rise to cross-linked polymers, bakelite.

Polyisoprene is natural rubber cis form its trans form is called gutta percha.

In inorganication, disulphide links are formed.

# Molecular wt. of Polymer : CAvg. molecular wt.





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Number avg. let  $n_1, n_2, n_3$  be no. of molecule chains having mass  $M_1, M_2, M_3$

$$\therefore \bar{M}_n = \frac{\sum n_i M_i}{\sum n_i}$$

$$M_w = \sum n_i M_i^2$$

$$M_D = \frac{\sum n_i M_i^3}{\sum n_i M_i^2}$$

$$\text{Poly DISPERSEITY INDEX} = \frac{M_w}{M_D}$$

PDI = 1

Monodisperse

Natural polymers

PDI > 1

Polydisperse

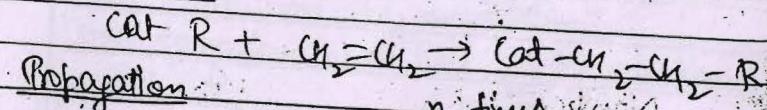
Synthetic polymers

of Coordination Polymerisation

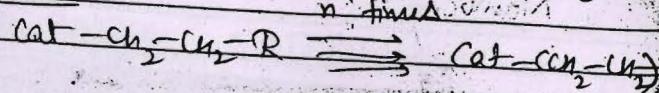
Ziegler - natta catalyst Cat - R  
( $R_3Al + TiCl_4$ ) R - Me, Et.

Working separately Ziegler & Natta developed it. Both received Nobel prize in 1965.

Initiation

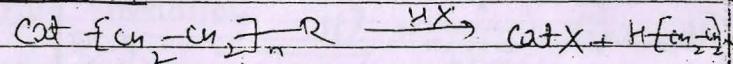


Propagation

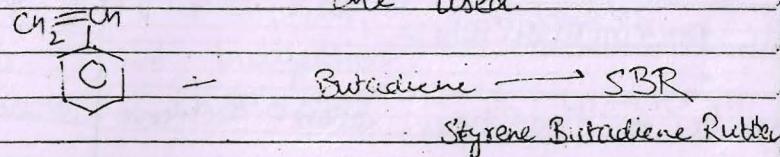


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Termination



# Co-polymerisation : Two diff. monomers are used.



In vulcanisation 2-3% S is added  
If 32% S is added, carbonite is formed

Kevlar

Monomers - Terephthalic acid + 1,4 dianime benzene

Nomax

Monomers - Metaphthalic acid + 1,3 dianimobenzene

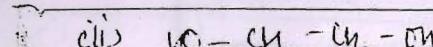
Poly carbonates.

Practice Problems

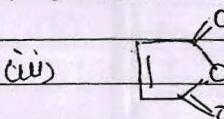
POLYMERS

Q.1 What is the functionality of following monomer in reaction with

- (a) styrene      (b) adipic acid
- (i)  $\text{H}_2\text{C}=\text{CH}-\text{CH}_2-\text{CH}_2$
- Am (a) 2      (b) 1



Ans (a) 0      (b) 2



Ans (a) 2      (b) 0

Q.2 A particular sample of polymer contains 200 molecules with molecular mass  $10^3$ , 300 with  $10^4$  and 500 with  $10^5$ . Find  $\bar{M}_w$ ,  $\bar{M}_n$  and PDI.

$$\bar{M}_w = \frac{\sum N_i M_i^2}{\sum N_i M_i}$$

$$= \frac{200 \times 10^6 + 300 \times 10^8 + 500 \times 10^{10}}{200 \times 10^3 + 300 \times 10^4 + 500 \times 10^5}$$

$$= \frac{200 + 300 \times 10^3 + 500 \times 10^5}{2 + 30 + 500}$$

$$= 56302000 = 9.45 \times 10^4$$

$$\bar{M}_n = \frac{\sum N_i M_i}{532}$$

$$= \frac{200 \times 10^3 + 300 \times 10^4 + 500 \times 10^5}{10000}$$

$$= 200 + 300 + 500$$

$$= 5.32 \times 10^3$$

$$\text{PDI} = \frac{\bar{M}_w}{\bar{M}_n} = \frac{9.45}{5.32} = 1.78$$

Q.3 Calculate end gp. content of polystyrene if molecular wt. 1,50,000 assuming phenyl gp. is to be present at both ends.

Ans. Mass of Ph. gp. = 77.

$$\therefore \text{End gp. content} = \frac{(2 \times 77 \times 100)}{150000}$$

$$= 10.27 \times 10^{-2} \text{ or } 0.012$$

\* Q.4 Styrene monomer having 0.02% by wt. benzoyl peroxide initiator was reacted until all the initiator was consumed. If 22% monomer is unreacted, calculate avg. degree of polymerisation of polymer. Assume 100% efficiency of initiator & coupling terminus.

Ans. Molecular mass of styrene = 105.

$$\text{At Benzoyl per.} = 242$$

Let  $x$  gm styrene.

$$\therefore \text{wt. of benzoyl peroxide} = 2 \times 10^{-4} x$$

$$\frac{78}{100} \times \text{gm styrene} \rightarrow 2 \times 10^{-4} \times \text{gm b.p.}$$

No. of polymer molecules formed =  $\frac{x}{105}$  mol. to pmol.

$$= \frac{x}{105} \times \frac{2 \times 10^{-4}}{242} x$$

$$\text{No. of monomer molecules} = \frac{78}{100} \times \frac{100 \times 10^3}{105}$$

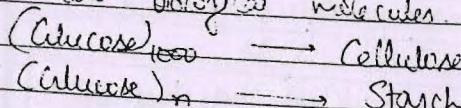
$$\text{Degree of poly.} = \frac{N_{\text{pmol}}}{N_{\text{poly}}} = \frac{18200}{2} = 9100$$

## BIOMOLECULES

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Carbohydrates, protein, lipids, amino acids etc.  
These are biological molecules.



$n \gg 1000$

↳ Carbohydrates - Polyhydroxy aldehydes & ketones or their compounds which produce such as units on hydrolysis.

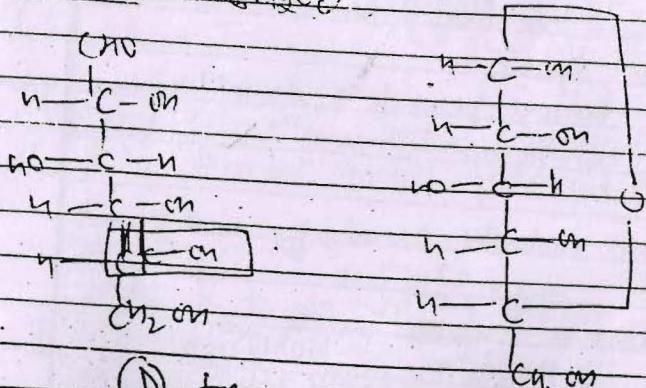
- Mono. (don't hydrolyse)
- Oligosaccharide (hydrolyse to 2-9)
- Poly (hydrolyse to  $> 9$ )

Blood Sugar - Glucose

Table Sugar - Sucrose

Milk Sugar - Lactose

Glucose + ( $\text{C}_2\text{H}_5\text{OH}_2$ )

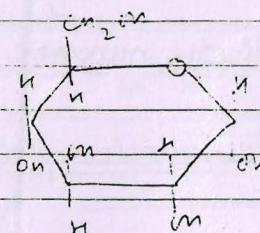


(D)-form

w.r.t. glycoside

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### Basics

Purine

Pyrimidines

A, G

C, T, U

Base + Sugar  $\longrightarrow$  Nucleoside

Base + Sugar + phosphate  $\longrightarrow$  Nucleotide

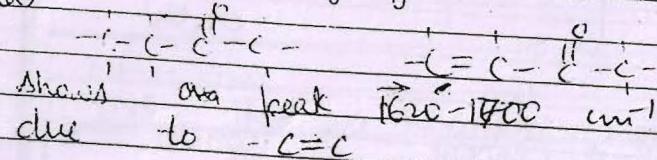
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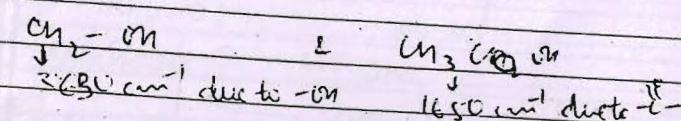
### SPECTROSCOPY

- Q.1. How will you diff. following on basis of IR  
 (a) Ethyl methyl ketone & vinyl methyl ketone  
 (b) methanol & ethanoic acid  
 (c) 2-hydroxy & 3-hydroxy benzoic acids

Ans (a)



(b)



(c)



- Q.2. An organic compound having molecular formula  $\text{C}_3\text{H}_6\text{O}$  shows an IR band at  $2950 \text{ cm}^{-1}$  and none near  $3300 + 1720 \text{ cm}^{-1}$ . Deduce structure.

Ans. Aliphatic C-H No acetylenic C-H, no aldehyde or ketone, no alcohol.  
 i. Must be an ~~ether~~ ether  
 $n-\text{C---O---C---n}$

### TITRIMETRY

- Q.1. 0.28 g  $\text{CaCO}_3$  was dissolved in  $\text{HCl A soln}$  made up 1 litr with distilled water. 100 ml of this soln reqd 28 ml EDTA on titration.  
 10 ml hard water sample reqd 35 ml of same EDTA soln 100 ml of boiled & cooled sample reqd 10 ml EDTA soln. find hardness with temp & permanet.

Ans. Normality of standard soln =  $\frac{0.28 \times 2}{100}$   
 $= 0.0056 \text{ N}$

Norm.  $N_{\text{std}} V_{\text{std. soln}} = N_{\text{EDTA}} V_{\text{EDTA}}$   
 $0.0056 \times 100 = N_{\text{EDTA}} \times 28$

$N_{\text{EDTA}} = \frac{0.56}{28} = 0.02 \text{ N}$

Now,  
 $N_{\text{EDTA}} V_{\text{EDTA}} = \frac{\text{Norm. sample}}{\text{Water sample}} \times \frac{V_{\text{water sample}}}{V_{\text{EDTA sample}}}$   
 $N_{\text{water sample}} = \frac{0.02 \times 35}{100} = 0.007$

Strength =  $N \times E_g \text{ wt} = 0.35 \text{ gm ff}^{-1}$   
 $= [350 \text{ ppm}] \text{ Total hardness}$

i. Permanet hardness =  $\frac{100 \times 50 \times 0.02 \times 10}{100} = 100 \text{ ppm}$   
 ii. Temp. hardness =  $\frac{100 \times 100}{250} = 40 \text{ ppm}$

- Q.2. 50 ml of sample water required 5 ml  $\text{N HCl}$  using Me but didn't give any precipitate with  $\text{HgCl}_2$ . Express alkalinity in ppt. & also its type.

1.  $\Phi = 0$   
2.  $\Phi = \frac{1}{2} M$   
3.  $\Phi < \frac{1}{2} M$



P - HPh  
M - Methyl  
Orange

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S.M. Alkalinity is due to  $\text{HCO}_3^-$  ion only as it gives colour only with Me.

$$\text{Also } N.V. = N_2 V_1$$

$$N_{\text{sample}} = 5 \times N_{\text{standard}} \times \frac{10}{50} \times 82.16$$

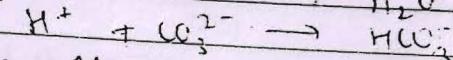
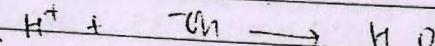
$$= 0.002 N$$

$$\text{Strength} = 0.002 N \times 5 = 0.01 \text{ gm/l}$$

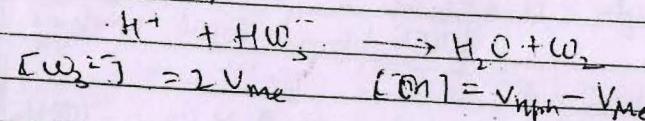
$$= 10 \text{ ppm}$$

Q.3. 200 ml water sample req. 20 ml  $N_2^{\text{HCO}_3}$  HU using Me as indicator. Another 200 ml  $N_2^{\text{HCO}_3}$  of same sample req. 8 ml  $N_2^{\text{HCO}_3}$  HU using HPh as indicator. Calculate alkalinity in terms of  $\text{CaCO}_3$ .

With HPh.



With Me,

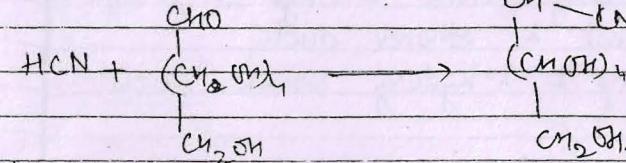


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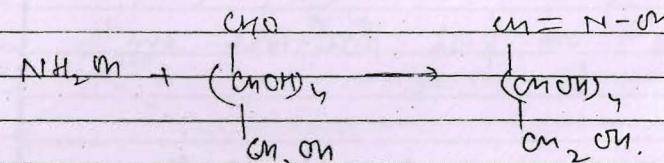
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Biomolecules  
Chemical Properties of glucose

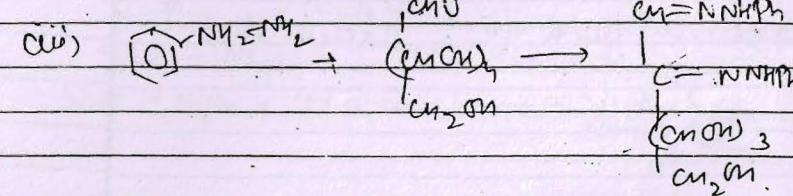
(i) Reactn with HCN



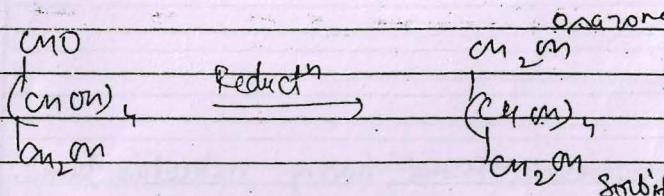
(ii)



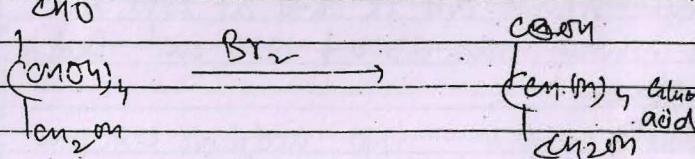
(iii)



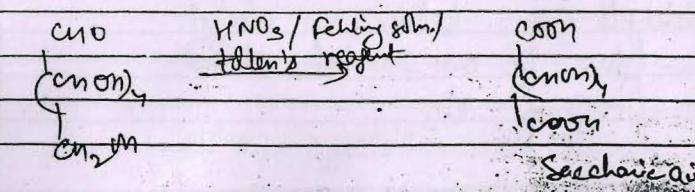
(iv)



(v)



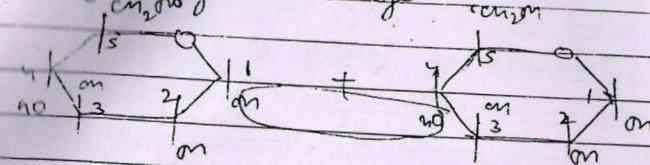
(vi)



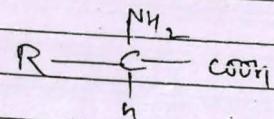
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CELLULOSE +  $(C_6H_{10}O_5)_n$

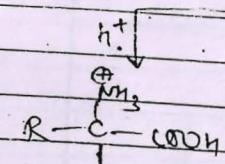
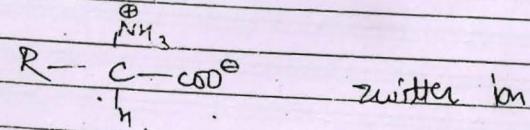
1,4 glycosidic linkage



# PROTEINS : These are polyamides. Monomeric unit is  $\alpha$ -amino acid.

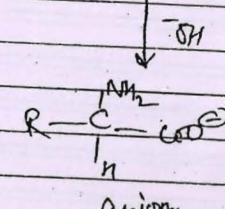


At equivalence pH,  $\alpha$ -amino acids form zwitter ions.



cation

moves towards cathode



anion  
moves towards anode

At isoelectric point, mobility and solubility of zwitter ion is minimum. Protein can

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be extracted only at this point.

\* All naturally occurring sugars belong to D series  
amino acids belong to L series.

for neutral amino acids, I.P. < 7

e.g., 6.1 for glycine  $R=H$   
alanine  $R=CH_3$

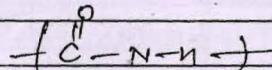
for acidic amino acids, I.P. 3.2-3.8

e.g., Aspartic  $R=CH_2COOH$   
Glutamic  $R=CH_2CH_2COOH$

for basic amino acids I.P. 10.8

e.g.,

# PEPTIDE BOND (-CONH-)

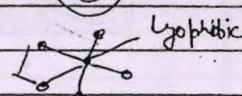
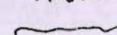


# Classification of proteins:

(a) On basis of structure.

Fibrous

Globular



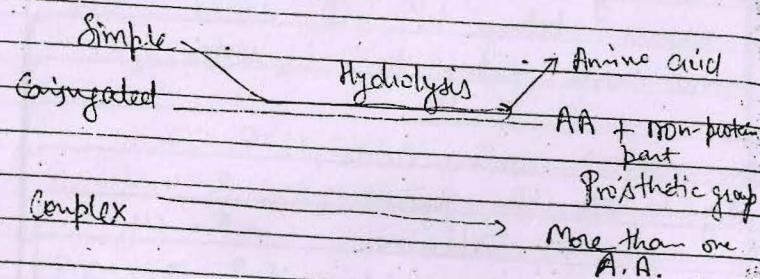
Globular

- Globular proteins are water soluble

Hydrophilic part

(iv)

(b) On basis of chemical composition  
Simple, conjugated, derived.



## # NUCLEIC ACIDS

Bases - Purine, Pyrimidine  
AG, CTU

Sugar - Ribose, deoxyribose  
A = T C = G

Higher GC content provides higher m.pt  
to a D.N.A.

higher GC content

