POC-I

pk_a :- HI > HBr > HCl > H_2SO_4 > HNO_3 > CH_3COOH > H_2S > HCN
−10 −9 −8/−7 −5 −2 ≈4 8 10
HF
3.4

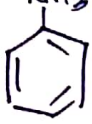
Elemental Analysis

(I) Qualitative Analysis

↳ Used to detect presence of element or elements.

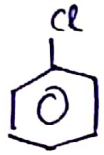
Ex:- C, H, N, S, P, O, −X:

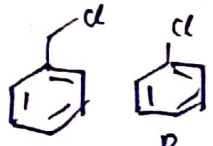
Ex:- $NH_3^{\oplus}Cl^{\ominus}$

1)  $\xrightarrow{AgNO_3}$ AgCl↓ white ppt. $\xrightarrow[(or)\ aq.NH_3]{NH_4OH}$ $[Ag(NH_3)_2]^+$ completely soluble due to complex formation

2) Hydrazine Hydro bromide $NH_2-NH_3^{\oplus}\ Br^{\ominus}$ $\xrightarrow{AgNO_3}$ AgBr↓ pale yellow ppt. $\xrightarrow[(or)\ aq.NH_3]{NH_4OH}$ $[Ag(NH_3)_2]^+$ partially soluble due to less ionic character.

3) $NH_4^{\oplus}I^{\ominus}$ $\xrightarrow{AgNO_3}$ AgI↓ yellow ppt. $\xrightarrow[(or)\ aq.NH_3]{NH_4OH}$ Insoluble

4)  $\xrightarrow{AgNO_3}$ No. ppt. formation.

Q. 
A B

Two compds, individually treated with aq. NaOH, then little amount of HNO_3 is added. Then $AgNO_3$ is added, one gives ppt while other doesn't give ppt.

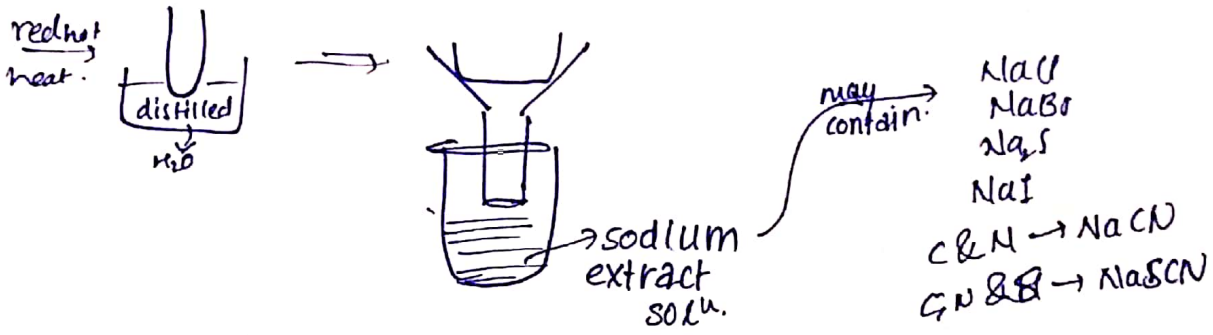
A → S_N2 → thus ppt. is formed.

B → NO S_N

* Lassaigne's test :- preparation of

Used for preparation of ion from corresponding covalent molecules.

procedure :-
- → Take Ignition tube with ~~given compnd~~ Na metal
- → Heat it under flame.
- → Then cool the test tube and fill it with organic compnd. Then heat it upto red hot condition.



Test (I) – for Halides

(i) sodium extract + $AgNO_3$ → $AgX\downarrow$
color may be – white
– pale yellow
– yellow

$Ag_2S\downarrow$ (if S is present)

AgCN↓ (if C & N are present)

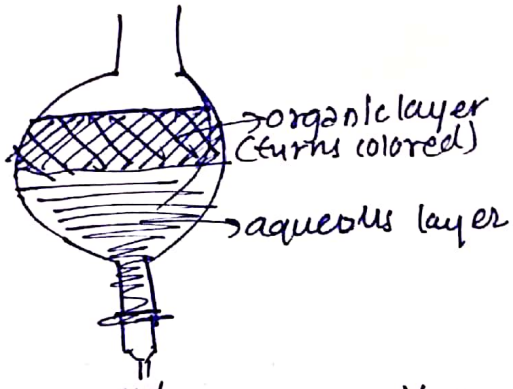
We have to add HNO_3 because the interfering ions may react with $AgNO_3$ to give other ppts. thus making detection of AgX ppt. difficult.

$$HNO_3 + NaCN \longrightarrow H-C\equiv N\uparrow \quad (P_{Ka}=10)$$
$$Na_2S + HNO_3 \rightarrow H_2S\uparrow \quad (P_{KA}=8)$$

(ii) Layer's test

Sodium extract $\xrightarrow[\text{with } Cl_2]{CCl_4/CHCl_3/Et_2O} X_2$

NaBr $\xrightarrow{Cl_2}$ Br_2 (reddish brown)
NaI $\xrightarrow{Cl_2}$ I_2 (violet)



(iii) Beilstein's test

With a loop of copper wire take organic compnd and keep it on flame, the ~~the~~ compound burns with bluish green flame.

Due to formation of CuX_2 & this color is observed, here X ≠ F. because CuF_2 is non-volatile. Thus used for detection of Cl, Br, I.

some other compnds giving this test:-

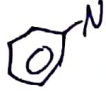
★  , $NH_2-\overset{S}{\overset{\|}{C}}-NH_2$, also show bluish green flame without halogen.

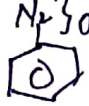
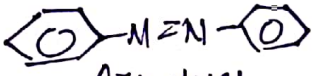
Test (2) – for Nitrogen

NaCN,
Sodium extract $\xrightarrow{FeSO_4}$ $Na_4^{+1}[Fe^{II}(CN)_6]^{-4}$ $\xrightarrow[\text{with } H_2SO_4]{\text{acidified}}$ $\xrightarrow[\text{exposed to air}]{\text{add } FeCl_3}$ $Fe^{III}_4[Fe^{II}(CN)_6]_3$
sol^n prussian blue.

Important pt. :–

(i) NH_3, $NaNO_3$, NH_2-NH_2, NH_2-OH, etc. doesn't give prussian blue due to absence of carbon.

(ii) KCN,  ,  ,  , NH_2CONH_2, etc. give prussian blue.

(iii) 
diazonium salts, 
Azo dyes or compnds. , diazomethane, won't give prussian blue

because on heating they decompose to give $N_2\uparrow$ avoiding formation of $CN^{\ominus}$ ion and thereby don't give this test.

(iv) If organic compn possess C, N and S, then formation of NaSCN takes place, it doesn't give prussian blue color.

(v) If test is carried with excess of Na, then, if organic compn contains both 'N' and 's', shows characteristic test for both CN^- and S^{2-}.

$NaSCN \xrightarrow[\text{excess}]{Na} NaCN + Na_2S$.

Test (3) – for sulphur

(i) Sodium extract sol^n $\xrightarrow{Pb(OAc)_2}$ $PbS\downarrow$ black ppt.

(ii) Sodium extract sol^n – $\xrightarrow{\text{sodium nitro prusside.}}$ violet color due to formation of sodium thio nitro prusside.
$Na_2[Fe^{+2}(CN)_5(NO^{+1})]^{-2}$

Test (4) – for both N and 's'. [i.e for (SCN^-)]

Sodium extract sol^n (limited Na) $\xrightarrow{FeCl_3}$ $[Fe(CSCN)]^{2+}$ Blood red coloration.

Test for 'P'
(5)

Organic compnd $\xrightarrow[\text{(or) } HNO_3]{Na_2O_2}$ H_3PO_4 $\xrightarrow[\text{Ammonium molybdate}]{(NH_4)_2MoO_4}$ $(NH_4)_3PO_4 \cdot 12MoO_3$ (yellow ppt) Ammonium phospho molybdate.

H_3PO_4 $\xrightarrow{\text{magnesium mixture } (MgCl_2 + NH_4Cl + NH_3)}$ $Mg(NH_4)PO_4$ white ppt

$\downarrow \Delta$

$Mg_2P_2O_7$
Magnesium pyro phosphate.

Test (6) – for 'C' & 'H'

Leibig's method

organic compnd $\xrightarrow[\Delta]{CuO}$ $CO_2 + H_2O$ $\xrightarrow{\text{anhydrous } CuSO_4}$ $CuSO_4 \cdot 5H_2O$ (Blue color)

CO_2 $\xrightarrow{\text{lime water}}$ $CaCO_3 \downarrow$ (milky white color)

Test (7) – for 'O'

No method is available.

II Quantitative Analysis

(1) 'C' and 'H'

Leibig's Method

organic compounds (W gms) $\xrightarrow[\Delta]{CuO}$ $CO_2 + H_2O$

$CO_2 \xrightarrow{KOH} K_2CO_3$ (W_2 gms)

$H_2O \xrightarrow{\text{Anhyd. } CaCl_2}$ (W_1 gms)

$$\% H = \frac{2}{18} \times \frac{W_1}{W} \times 100$$

$$\% C = \frac{12}{44} \times \frac{W_2}{W} \times 100$$

(2) Calculation of 'N'

* KJELDAHL'S METHOD.

org. compd. (W gms) $\xrightarrow[\Delta]{H_2SO_4}$ $(NH_4)_2SO_4$ $\xrightarrow{Base}$ NH_3

NH_3 $\xrightarrow{\text{conc. } H^+ \text{ (excess) (with known concentration)}}$ $(NH_4^+ + H^+)$

$(NH_4^+ + H^+)$ $\xleftarrow{\text{dilute base (with known concentration)}}$

n_1 moles of H^+

n_2 moles of OH^-

$$\% N = \frac{(n_1 - n_2) \times 14 \times 100}{W}$$

→ This method is not applicable for following methods.

(i) compounds containing Nitro $-NO_2$ grps.

(ii) If compds containing azo grps $(-N=N-)$.

(iii) Pyridine containing rings.

* Duma's Method

org. compnd. (W gms) $\xrightarrow[\text{Atm of } CO_2]{(II)\ CuO}$ $N_2 + CO_2 + H_2O$ + oxides of N $\xrightarrow{\text{passed through copper gauze}}$ $N_2 + CO_2 + H_2O$

$N_2 + CO_2 + H_2O \xrightarrow{KOH} N_2 + H_2O + K_2CO_3$

↓ collect pure N_2.

$$\% N = \frac{PV}{RT} \times \frac{28}{W} \times 100$$

(3) for halogen

Carius Method

org compnd. (W gms) $\xrightarrow[2)\ AgNO_3]{1)\ con.\ HNO_3}$ AgX (W_1 gm)

$$\% Cl = \frac{35.5}{143.5} \times \frac{W_1}{W} \times 100$$

$$\% Br = \frac{80}{188} \times \frac{W_1}{W} \times 100$$

$$\% I = \frac{127}{235} \times \frac{W_1}{W} \times 100$$

(4) for 'S'

Carius Method

Org. comd. $\xrightarrow[HNO_3 / H_2SO_4]{Na_2O_2} H_2SO_4 \xrightarrow{BaCl_2} BaSO_4 \downarrow$

(W gms)

$\% S = \frac{32}{233} \times \frac{W_1}{W_2} \times 100$

(5) for 'P'

Org. comd $\xrightarrow{HNO_3} H_3PO_4 \xrightarrow{(NH_4)_2MoO_3} (NH_4)_3PO_4 \cdot 12MoO_3$

(W gms) (W gms)

↓ mg mixture

$Mg_2P_2O_7$

W_2 gms.

$\% P = \frac{31}{187} \times \frac{W_1}{W} \times 100$

$= \frac{62}{222} \times \frac{W_2}{W} \times 100$

III

purification methods of organic compnds

(1) Sublimation

→ camphor, benzoic acid, napthalene, anthracene, I_2, NH_4Cl, $HgCl_2$, etc.

(2) Crystallisation

→ solubility decreases as temp. decreases.

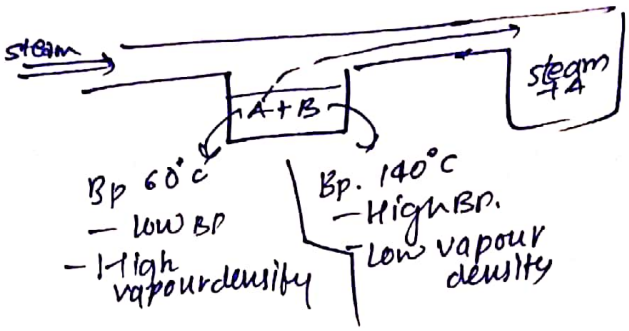
→ The soln ~~used~~ left over after crystallisation is called mother liquor.

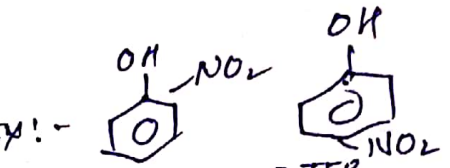
(3) Distillation :-

→ used for liquids.

→ If huge diff. is there in their B.p $\geq 40^\circ C$ conduct distillation. And if diff. in B.p is $\leq 40^\circ C$ conduct fractional distillation.

Steam Distillation





INTRA
Low BP.
Insoluble in $H_2O_{(l)}$.
but soluble in $H_{2(g)}$.

INTER
High BP

Vacuum Distillation

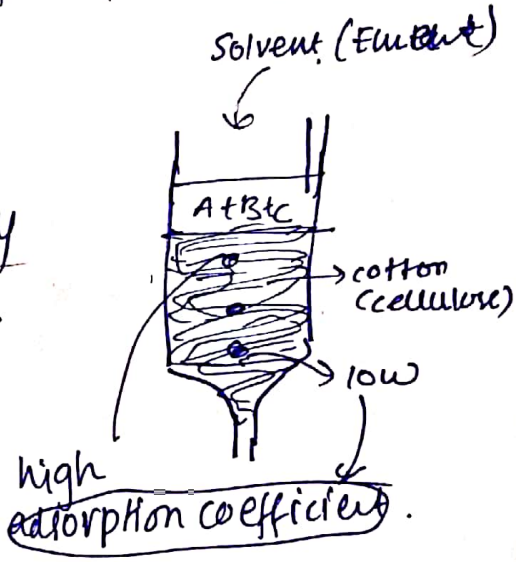
→ Distillation under reduced ~~notes~~ pressure.
→ glycerine, cane sugar purification.

chromatography

→ Advanced technique best method for separation of mixture and purification of mixture.

(A) Adsorption chromatography :-

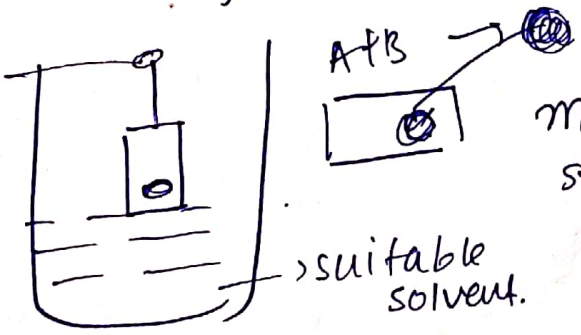
i) Column chromatography



A, B, C have diff. adsorption coefficient.

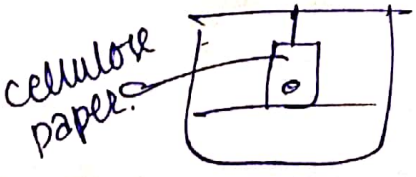
stationary phase - solid
mobile phase - liquid.

(ii) Thin layer chromatography



mobile phase :- liquid.
stationary phase :- solid.

Partition chromatography



mobile phase :- liq.
Stationary phase :- (liq)