Boiling point - 120°C.

Lab Normality - 11.3

molecular weight - 36.

Importance of HCl in decomposition analysis:-

→ HCl is a basic solvent in the analysis of ores and minerals.

→ It's advantages are easy volatality, solubility of it's salt's in water and weak reducing effect on dilution.

→ In decomposing samples with HCl the chlorides of Germinium(IV), arsenic(III), stelbium(III), Sn(IV), Se(IV), Hg(II) are observed.

→ It has complexing ability with majority of metals like $[NiCl_4]^{2-}$, $[FeCl_6]^{3-}$, $[FeCl_6]^{4-}$ etc.

→ Most important ores areanionic complexes with maximum coordination number and neutral complexes.

→ The anionic character of the individual complex, chloride ion have been made use for the seperation of metals on anion exchange resin.

→ The formation of the chloride complexes many metals has also utilising extraction

Vessels:-

→ In decomposition with Hcl it is usual to use glass vessels.

→ It platinum vessels also used

→ 37% of Hcl effects gold, Rhodium, vanadium, palladium the same way effects on platinum also.

→ Hcl decomposes hydrated oxides more easily than anhydrous oxides.

→ complicated uranyl carbonates dissolve completely in 5% Hcl

→ To decompose flow fluoro carbonates of rare earth metals easily by Hcl.
ex:- parasite

→ Heavy metals like carbonates, malachite, azurite, smithsonite, cerussite, # zaratite are easily soluble in Hcl.

→ The most suitable decomposition is prilimin dissolution of limestone is (1:1 dil. Hcl).

→ The seperated insoluble residue is then decomposition by mixture of ($HF + HNO_3$), after volatalisation of SiF_4. The residue is dissolved in concentrated Hcl.

→ calcite, Argonite dissolved very easily at room temprature in dilute (1:3) HCl.

→ similarly Natural barium carbonates (witherite) and strancium carbonate (strontianite) decompose easily

$2LiCO_3 + 2HCl \longrightarrow 2LiCl + H_2O + CO_2$

$Na_2CO_3 + HCl \longrightarrow NaCl + H_2O + CO_2$

$Na_2S_2O_3 + 3HCl \longrightarrow 2NaCl + SO_2 + S\uparrow + H_2O$

$mgCO_3 + 2HCl \longrightarrow mgCl_2 + H_2O + O_2$

$GeO_2 + 4HCl \longrightarrow GeCl_4 + 2H_2O$

$SnO_2 + 4HCl \longrightarrow SnCl_4 + 2H_2O$

$PbO_2 + 4HCl \longrightarrow PbCl_2 + Cl_2 + 2H_2O$

$Sb_2O_3 + 6HCl \longrightarrow 2SbCl_3 + 3H_2O$

$CaOCl_2 + 2HCl \longrightarrow CaCl_2 + H_2O + Cl_2$

$CuSO_4 + 2HCl \longrightarrow CuCl_2 + H_2SO_4$

$ZnCO_3 + 2HCl \longrightarrow ZnCl_2 + CO_2 + H_2O$

$Hg_2(NO_3)_2 + 2HCl \longrightarrow Hg_2Cl_2 + 2HNO_3$

$Fe_2O_3 + 6HCl \longrightarrow 2FeCl_3 + 3H_2O$.

18/7/19

→ Decomposition with HNO_3: Molecular wt - 63.0
Lab normality = 11.5N B.P - 121°C

→ HNO_3 dilute as well as concentrated is powerful solvent for a number of minerals especially for sulphides and phosphates.

→ It is strong oxidant it can be oxidises sulphides to sulphates.

→ Antimony and Tin sulpho salts are converted to the respective metals acids of the higher oxidation states.

→ Sulphur and sulphides are must be oxidised at room temperature.

→ HNO_3 dissolves the majority of metals occur in nature with the exception of gold and platinum.

→ In the general alloys of silver with palladi are very soluble, their solubility is less if Iridium, Rhodium and platinum are present, platinum is difficult to dissolves even if present in a ratio to silver of 1:10, the dissolution must be repeated several times.

→ The majority of copper sulphide and oxides ores are soluble in HNO_3

→ If the components which volatiles at

elevated temprature (Hg, Se, Te, Zn, mo, As, Sb) decomposing with HNO_3

→ Zinc and Cadmium sulphides are easily decomposed with HNO_3

→ Iron disulphides which dissolves in HNO_3

→ while the cusic form pyrite decomposes without a residue in con. HNO_3

→ dil. HNO_3 decomposed some antimony and tin sulphonats at room temprature

→ The molbdynum disulphides, molbdenite is decomposed by conc. HNO_3.

$$BaCO_3 + 2HNO_3 \longrightarrow Ba(NO_3)_2 + CO_2 + H_2O$$

$$PbO + 2HNO_3 \longrightarrow Pb(NO_3)_2 + H_2O$$

Lead oxide — Lead Nitrate

$$Pb_3O_4 + 4HNO_3 \longrightarrow 2Pb(NO_3)_2 + PbO_2$$

$$CaO + 2HNO_3 \longrightarrow Ca(NO_3)_2 + H_2O$$

$$Na_2CO_3 + 2HNO_3 \longrightarrow 2NaNO_3 + H_2O + CO_2$$

Sodium carbonate

$$CaCO_3 + 2HNO_3 \longrightarrow Ca(NO_3)_2 + H_2O + CO_2$$

Calcium Carbonate

$$CuO + 2HNO_3 \longrightarrow Cu(NO_3)_2 + H_2O$$

→ Decomposition with HF:- Lab normality - 26.5N molecular weight - 20.1; Boiling point - 112°C.

→ HF as monobychiac acid form a single series of hydrofluorides are co-ordination compounds with Hydrogen as the central atom of the complex.

→ The F^- anion form complexes mostly of medium strength but it has great afinity for some ions. Thus characteristic fluoride complexes are formed with beryllium (BeF^-) Boron type (BF_4^-), Scandium, Aluminium, Iron and some other evansition elements in the presence of an excess of the fluoride ion.

→ In ion exchange chromatography fluoride complexes are used mainly for the seperation of Niobium, tantalum, titatinum and Zirconium.

→ Formation of Fluoride complexes has also been utilised for effective seperation As^{III}, As^{V}, Sb^{III}, V^{III}, Ge^{IV}, V^{V}, W^{VI}, Se^{IV}, Te^{IV}, Mo^{VI} and Ta^{V} are extracted by large degree.

Vessles:-

→ HF may be easily stored in polythene vessles which are not attacked by 40% HF easily even at 100°C.

→ Teflon- polytetra fluoro ethylene is exceptionally resistant to the effect of chemicals in wide temprature range. It canbe used upto 250°C. It loses strength a wide temprature and complete depolymerization occurs at 400-450°C.

→ Teflon is very useful for work with HF which does not attack it at all.

→ A plastic equally resistant but more easily worked is poly tri fluoro ethylene - Hostaflon (Fluorothene)

→ Gold, Rhodium, Irridium, palladium and platinum are stable in 40% HF upto the BP of the acid.

→ It reacts with silicon oxide or silicates forming the very unstable volatile SiF_4 which volatiles when heated.

→ since the fluorides of some elements especially of the IV group of the periodic system (Sn, Ti, Zr, Ge) are volatile.

→ Iron, Aluminium and titanium and sulphates formed in the course of decomposition with HF and H_2SO_4 may be converted to insoluble oxides by heating at 650°C.

$$SiO_2 + H_2F_2 \longrightarrow SiF_4 + H_2O$$

$$SiF_4 + H_2F_2 \longrightarrow H_2SiF_6$$
(Hydro fluorsilicic acid)

$$Na_2SiO_3 + 3H_2F_2 \longrightarrow Na_2SiF_6 + 3H_2O$$

$$Cl_2O_6 + HF \longrightarrow HClO_4 + ClO_2F$$

$$XeF_6 + HF \longrightarrow [XeF_5^-] + [HF_2]^-$$

9/11/4

Decomposition with H_2SO_4:-

Lab normality - 36 N

molecular weight - 98.08

Boiling point - 338°C.

→ Hot concentrated H_2SO_4 as a weak oxidising effect in the dissolution process. Generally reduce to sulphurous acid sometimes this reduction contineous forming sulphur (or) H_2S.

→ H_2SO_4 oxidised not only inmetals but other substances also.

Ex:- Sulphur and cole, which are oxidised with evaluation of SO_2.

→ Arsenic and Antimony and tin are oxidised to trivalent to tetra valent state.

→ The sulphuric acid anions behaves as a di basic ligand forming complex of medium strength with tri and tetra valent metal cations.

Vessles:-

→ when samples of being decomposed with H_2SO_4 in platinum vessles at elevated temprature

→ upto 250° gold is relatively stable with respect to the effect of 96% of H_2SO_4

→ In conc. H_2SO_4 poly ethylene is also stable upto 50°c.

→ on the otherhand Teflon is resistant upto 300c.

→ It is efficient solvent for minerals, ores and decomposition of sulphides, fluro silicate, Niyobium, tantalum, Titanium, Zirconium and gold

→ H_2SO_4 is a solvent the decomposition of rare earth phosphates, loparaites, hatchett-lite, pyrochlores and uranothorianite dissolves easily in H_2SO_4.

→ H_2SO_4 is used for oxigen conteining mineral halide(fluorides), celerium, Telurium

→ con. H_2SO_4 used for decompose sulphides, antimony ores, poly metallic ores and thallium.

$$BaO_2 \cdot 8H_2O + H_2SO_4 \longrightarrow BaSO_4 + H_2O_2 + 8H_2O$$

$$Ba_3(PO_4)_2 + 3H_2SO_4 \longrightarrow 3BaSO_4 + 2H_3PO_4$$

$$2LiAl(SiO_3)_2 + 4H_2SO_4 \longrightarrow Li_2SO_4 + Al_2(SO_4)_3 + 4H_2SiO_3$$

$BaCl_2 + H_2SO_4 \longrightarrow BaSO_4 + 2HCl$

$BaS + H_2SO_4 \longrightarrow BaSO_4 + H_2S$

$FeS + H_2SO_4 \longrightarrow FeSO_4 + H_2S$

$Na_2SO_3 + H_2SO_4 \longrightarrow Na_2SO_4 + SO_2 + S + H_2O$

$BeO + H_2SO_4 \longrightarrow BeSO_4 + H_2O$

$MgO + H_2SO_4 \longrightarrow MgSO_4 + H_2O$

$CaCl_2 + H_2SO_4 \longrightarrow CaSO_4 + 2HCl$

$2B + 3H_2SO_4 \longrightarrow 2H_3BO_3 + 3H_2$

$Ti_2O_3 + 3H_2SO_4 \longrightarrow Ti_2(SO_4)_3 + 3H_2O.$

$Na_2B_4O_7 + H_2SO_4 \longrightarrow Na_2SO_4 + H_2B_4O_7$

$Al_2O_3 + 3H_2SO_4 \longrightarrow Al_2(SO_4)_3 + 3H_2O$

$K_2SO_4\,Al_2(SO_4)_3\,4Al(OH)_3 + 6H_2SO_4 \longrightarrow K_2SO_4 + 3Al_2(SO_4)_3 + 12H_2O$

$SnO + H_2SO_4 \longrightarrow SnSO_4 + H_2O.$

$K_4Fe(CN)_6 + 3H_2O \longrightarrow 2K_2SO_4 + FeSO_4 + 6HCN$

$$\begin{matrix} COOH \\ | \\ COOH \end{matrix} \xrightarrow[-H_2O]{H_2SO_4} CO + CO_2$$

$$ZnCO_3 + H_2SO_4 \longrightarrow ZnSO_4 + H_2O + CO_2$$

$$2Pb_3O_4 + 6H_2SO_4 \longrightarrow 6PbSO_4 + 6H_2O + O_2$$

$$2Cl\,Al(Si_2O_5)_2 + 4H_2SO_4 \longrightarrow Li_2SO_4 + Al_2(SO_4)_3 + 4H_2SiO_3 + 4SiO_2$$

24/9/14

Decomposition with perchloric acid ($HClO_4$)

molecular weight - 100.47

Boiling point - 203°c

Lab normality - 12 N

→ The properties of perchloric is very similary to those of H_2SO_4, compare to the other mineral acids, $HClO_4$ as a high boiling point.

→ $HClO_4$ is frequently used to precipitate silisic acid.

→ $HClO_4$ has been formed especially used for decomposition and the determination of silicon dioxide in calisite, dolamite, and magnisite.

→ when the samples contain organic substance it is first ignited at 500°c and then decompose with perchloric acid.

→ For the direct decomposition of natural phosphates containing organic compound, a miature of conc. HNO_3 and $HClO_4$ is used.

→ decomposition with $HClO_4$ is the advantage for the determination of bismuth in lead minerals.

→ Dolamite, alunite, calcinite, aluminium hydroxy sulphates are easily dissolved in perchloric acid.

→ A mixture of $HClO_4$, H_2SO_4 and HCl is a very efficient solvent for Nickel and Iron ores.

$$Zn + 2HClO_4 \longrightarrow Zn(ClO_4)_2 + H_2$$

$$2HClO_4 + P_2O_5 \longrightarrow 2HPO_3 + Cl_2O_7.$$

vessles:-

Glass, Quartz vessles are not used because they can not stand at high temprature but different types of crucibles are used in these fusion mixtures.

vessle	Temprature
platinum	– 1772°c
Nickel	– 1453c
copper	– 1083.05°c
Irradium	– 2450°c
AKum(gold)	– 1063°c
silver	– 960°c

Fusion methods can be broadly classified into four alternative ways.

1. Alkaholic
1. Alkali Fusion : Na_2CO_3 , NaOH
2. Acedic Fusion : $NaHSO_4$, $Na_2S_2O_7$ (Sodium pyro sulphate)
3. oxidative Fusion : Na_2O_2 (Sodium peroxide). Sodium chlorate ($NaClO_3$)
4. Reductive Fusion: $Na_2CO_3 + Na_4BO_4$.

Alkali Fusion:- (with $Na_2(CO_3)$:-

It is frequently used anhydrous material melt at 858c.

$$Na_2CO_3 \rightleftharpoons Na_2O + CO_2$$

→ carbonate fusion in the presence of air has an oxidative effect on many element (sulphur, manganese, chromium).

→ anhydrous Na_2CO_3 generally used as Flux for the decomposition of silicate rocks and minerals.

→ other Beryllium minerals eg:- phenocite, bertaxite, helvite are easily decomposed by fusion with Na_2CO_3.

→ Decomposition with mixture of borax and Na_2CO_3 has also been found useful for the determination of trace elements in Zircon.

→ The seperation of sulphates from Barium, Strontium or calcium carbonate in the decomposition.

→ The poorly solublue thorium and rare earth phosphates from (monazite) decompose easily when fused with Na_2CO_3.

→ Fusion with Na_2CO_3 can't be used to determine Thallium

$MgCl_2 + Na_2CO_3 \longrightarrow MgCO_3 + 2NaCl$

$CaSO_4 + Na_2CO_3 \longrightarrow CaCO_3 + Na_2SO_4$

$Fe_2(SO_4)_3 + 3Na_2CO_3 \longrightarrow Fe_2(CO_3)_3 + 3Na_2SO_4$

$CaCl_2 + Na_2CO_3 \longrightarrow CaCO_3 + 2NaCl$

$SiO_2 + Na_2CO_3 \longrightarrow Na_2SiO_3 + CO_2\uparrow$

$Li_2SO_4 + Na_2CO_3 \longrightarrow Li_2CO_3 + Na_2SO_4$

$Fe_2O_3 + Na_2CO_3 \longrightarrow 2NaFeO_2 + CO_2$

$2\,Cu\,S_2O_3 + Na_2CO_3 \longrightarrow Cu_2S_2O_3 + Na_2S_4O_6$

$3\,Cu_2 \cdot S_2O_3 + 2\,Na_2S_2O_3 \longrightarrow Na_4[Cu_6[S_2O_3]_5]$

$Ca_2B_6O_{11} + 2\,Na_2CO_3 \longrightarrow Na_2B_4O_7 + 2NaBO_2 + 2CaCO_3$

$BaCl_2 + Na_2CO_3 \longrightarrow BaCO_3 + 2NaCl$

$Si + Na_2CO_3 \longrightarrow Na_2SiO_3 + C$

9/10/14

Fusion with NaOH:-

→ Absolutely pure NaOH melts at 328°C but its having contents of water and CO_2 the melting point is usually about 10°C lower, when red hot NaOH volatiles to large extent.

→ Fusion with NaOH is used for decompose quartz, silicates, glass making sand, clays, silicides and carbides of various metals, natural oxides, tungstanates, titanates, fluorides, some ores and rocks.

→ Fusion with NaOH used for a large no of minerals.

→ This fusion generally carried out at crucibles of Fe, Ni, silver (or) gold but never of platinum.

$$M-X + NaOH \xrightarrow[30-60\,min]{400-600^{\circ}C} M-OH + Na-X$$

↓

soluble in water (or) acid.

Reactions:-

$$Zn + 2NaOH \longrightarrow Na_2ZnO_2 + H_2\uparrow$$

$$2Al + 2NaOH \longrightarrow 2NaAlO_2 + 3H_2\uparrow$$

$$Sn + 2NaOH + H_2O \longrightarrow Na_2SnO_3 + 2H_2\uparrow$$

$$Pb + 2NaOH + H_2O \longrightarrow Na_2PbO_3 + 2H_2\uparrow$$

$Ni(NO_3)_2 + 2NaOH \longrightarrow Ni(OH)_2 + 2NaNO_3$

$FeSO_4 + 2NaOH \longrightarrow Fe(OH)_2 + Na_2SO_4$

$mnSO_4 + 2NaOH \longrightarrow mn(OH)_2 + Na_2SO_4$

$CuSO_4 + 2NaOH \longrightarrow Cu(OH)_2 + Na_2SO_4$

$PbO_2 + 2NaOH \longrightarrow Na_2PbO_3 + H_2O$

$CrCl_3 + 3NaOH \longrightarrow Cr(OH)_3 + 3NaCl$

$P_4 + 3NaOH + 3H_2O \longrightarrow 3NaH_2PO_2 + PH_3$

$SiO_2 + 2NaOH \longrightarrow Na_2SiO_3 + H_2O$

$SnCl_2 + 2NaOH \longrightarrow Sn(OH)_2 + 2NaCl.$

⇒ Acidic fusion:-

Fusion with $NaHSO_4$ and $Na_2S_2O_7$:-

→ This is very efficient method used for ores of aluminium, iron, Titanium, tungsten.

→ which are not soluble in acid (or) acid mixtures. for carring out this acidic fusion.

→ For this acedic fusion, silica (or) porcelin curcible are used

→ The amount of bi sulphate (or) pyro sulphate require for this fusion depends upon the sample, some times as high as 30 times of sample.

→ This acidic fusion requires a temprature of 600 - 700°C, Finally the melt is extracted in water and dilute H_2SO_4.

$$TiO_2 + 2NaHSO_4 \rightleftharpoons Ti(SO_4)_2 + Na_2O + H_2O$$

$$TiO_2 + 2Na_2S_2O_7 \rightleftharpoons Ti(SO_4)_2 + Na_2O$$

$$NaIO_3 + NaHSO_4 \rightleftharpoons Na_2SO_4 + HIO_3$$

14/10/14

Oxidative Fusion:-

Fusion reagent:- Sodium peroxide & sodium chloride

Temprature : At low

Crucibles : Ni, Fe, Si etc.

Silica crucibles are not used bcoz it reacts with Na_2O_2.

$$2SnO_2 + 2Na_2O_2 \longrightarrow 2Na_2SnO_3$$

$$2SiO_2 + 2Na_2O_2 \longrightarrow 2Na_2SiO_3 + O_2$$

$$ZnS + 4Na_2O_2 \longrightarrow ZnSO_4 + 4Na_2O$$

$$2Al_2O_3 + 2Na_2O_2 \longrightarrow 4NaAlO_2 + O_2$$

$$Cr_2O_3 + Na_2O_2 \longrightarrow 2NaCrO_4 + 3Na_2O$$

$$Al_2O_3 + NaClO_3 \longrightarrow NaClO_2 + NaCl$$

$$ZnS + NaClO_3 \longrightarrow ZnSO_4 + NaCl$$

$$PbS + NaClO_3 \longrightarrow PbSO_4 + NaCl$$

procedure:-

Na_2O_2 can used efficiently as fused reagent and sodium chlorate, when compare to the low tempratures, requirement of low temperature permit crucibles such as Ni, Fe, Si etc

→ However silica & alumina crucibles are not suitable used bcoz a small error

→ The amount of Fusion mixture needed is about 5 times the weight of the sample,

Na_2O_2 is a fused first and then sample is added in small fraction followed by fusion.

→ This fusion becomes complete at dull red hot and takes only 5-10 min.

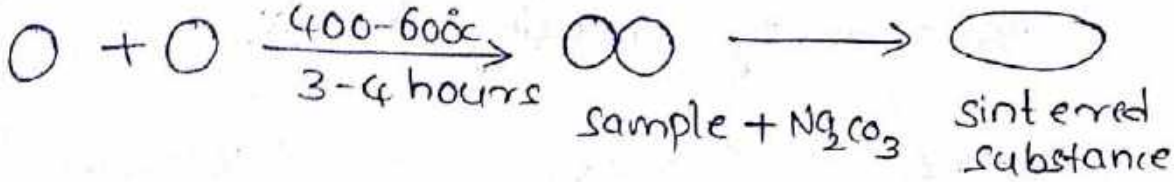
→ The melt is extracted with water (or) acid, the resulting solution is boiled to expell the hydrogen peroxide when the excess Na_2O_2 is dissolved in water, this method is particularly suitable for Tin, chromium ores.

Reductive fusion:- (Sodium carbonate + Sodium boroxide Na_4BO_4)

As well as Fusion with Sodium carbonate.

Sintering:-

$$\text{Sample} + Na_2CO_3 \xrightarrow[3-4 \text{ hours}]{400-600^\circ C} \text{Sintered substance.}$$



→ A process of where the sample mix throughly with selected substance in the ratio of 1:1 (or) 1:2 and that fine powder is heated to lower temprature for a long time during their heating process the fine powder will be slowly becomes a solid It is called sintering

→ The sintered material is reached with acids which gives soluble and insoluble materials which are later analysed for different constituents.

Decomposition by sintering with Na_2O_2:-

→ The majority of minerals may be decomposed by sintering with sodiumperoxide at low temprature.

→ Rocks, ores, magnetite, barytes, gypsum, quartz, fledspar, bauxite, decompose with Na_2O_2

→ To avoid the corrision problem of the vessles (or) crucibles, the sintering process using Na_2O_2 is carried out on pt- nickle crucible

the temprature should not kept at below 600.

→ Minerals like BrO, ZrO, Aluminate, are decompose efficiently even in the range of 200-300°C. by sintering process.

Reactions:- $SiO_2 + 2Na_2O_2 \longrightarrow Na_4SiO_4 + O_2$

$TiO_2 + 2Na_2O_2 \longrightarrow Na_4TiO_4 + O_2$

$Al_2Be_3Si_6O_{18} + 16Na_2O_2 \longrightarrow 6Na_4SiO_4 + 3Na_2BeO_2 + 2AlO_2 + 8O_2$

$2(FeOCr_2O_3) + 7Na_2O_2 \longrightarrow 2NaFeO_3 + 4Na_2CrO_4$

$2Cr(OH)_3 + 3Na_2O_2 \longrightarrow 2NaCrO_4 + 2NaOH + 2H_2O$

$MnSO_4 + 2Na_2O_2 \longrightarrow Na_2MnO_4 + Na_2SO_4$.

Decomposition by sintering with Alkali Hydroxide

Sintering process + Fusion NaOH Reactions

Decomposition by sintering with alkali carbon

Sintering process + Fusion with Na_2CO_3 Reacti

Difference b/w Fusion and sintering.

Fusion	Sintering
1. Fusion is a process where the sample is mixed with 10times (or) 25 times of Fusion mixture and heat up to 900-1200°C	1. Sintering process where the sample is mixed with almost 2-3 times fusion mixture heated up to 400-600°C.
2. metal silicates + Na_2CO_3 $\xrightarrow[900-1200°C]{30-60\ min}$ metal-CO_3 + Na-silicates ↓ metal + CO_2 ↑	2. m-silicates + Na_2CO_3 $\xrightarrow[400-600°C]{3-4\ hrs}$ m-CO_3 + Na-silicates ↓ metal + CO_2
3. Ex:- $CaB_6O_{11} + 2Na_2CO_3 \longrightarrow$ $Na_2B_4O_7 + 2NaBO_2 + CaCO_3$	3. $MnSO_4 + 2Na_2O_2 \longrightarrow$ $Na_2MnO_4 + Na_2SO_4$
4. Time of reaction is 30-60 mins	4. Time of reaction is 3-4 hours
5. Corrosion of vessels is very high (because of high temprature)	5. Corrosion of vessels is very low (because of low temprature)
6. Pt, Ir can be used an vessels.	6. Pt, Ir, Ni used as vessels
7. Size of the particle is high compared to Sintering process	7. The size of the particle is less compare to fusion.
8. We must use very pure fusion mixture (to avoid contamination)	8. We must use very pure fusion mixture to avoid contamination

9. In fusion Na_2CO_3, NaOH - Alkali fusion Na_2O_2, $NaClO_3$ - oxidative fusion Na_2CO_3, $Na_4B_2O_4$ - Reductive fusion.	9. In sintering Na_2CO_3 NaOH, Na_2O_2, $NaClO_3$, $Na_2CO_3 + Na_4B_2O_4$.
10. High temprature is required	10. low temprature is required
11. pressure is not applied	11. pressure is applied
12. The substance melts	12. The substance do not melts.
13. chemical reaction takes place.	13. chemical reaction don't takes place
14. The composition of substance changes.	14. The composition of substance dont change
15. New compounds are formed.	15. New compounds are not formed.
16. After the process the substance reached with water (or) acid.	16. After the process the substance reached with water (or) acid.

16/10/24

**Recrystallisation:-

Solid samples (Aromatic) are isolated from organic reactions they are usually contaminated the small amount of other components. (impurities).

→ which are produced along with desired product.

→ The purification of impure crystaline compounds is usually effected by crystalisation from a suitable solvent (or) mix of solvents.

→ The purification of solids by crystalisation is based on differences in their solubility in a given solvent (or) mix of solvents.

crystalisation process:-

Dissolve the impure substance in some suitable solvent nearly boilingpoint.

→ Filtering the hot solution from particles of insoluble material and dust

→ Allowing the hot, an cooled solution causing dissolved substance to crystalise out.

→ seperating the crystals from the mother solution (or) mother nuclear.

→ The resulting solvent after drying is tested for purity, if form impure in again recrystalised from fresh solvent.

→ The process is repeated untill the pure compound is obtained, but melting point is unchanged.

Solvents for recrystalisation:-

To compare high solvent power and low solvent power for the substance to be purified at a labaratory temprature is below (or) above

→ It should dissolve impurities readily are heated to only a small extent

→ It should yield well formed crystals. ~~are~~ of the purified compound.

→ It should capable of easy removal from the crystals of the purified compound.

Common solvents		Boiling point [°c]
water	—	100°c
CH_3OH (methanol)	—	64.5°c
Ethanol	—	78°c
Industrial spirit	—	77-81°c
rectified spirit	—	78°c
	—	56°c

Ethyl acetate	—	78°C
CH_3COOH	—	118°C
dioxime	—	101°C
Cyclo hexane	—	81°C
Benzene	—	80°C
CCl_4	—	77°C
$CHCl_3$	—	61°C
dichloroethane CH_2Cl_2	—	41°C
Di ethyl ether.	—	35°C
Light petroliem	—	40-60°C

Ultrasonic decomposition:-

→ In the decomposition of sample (or) dissolve the sample using ultrasonic wave which is frequently less than 20 Hz.

→ Ultrasonic energy has been used for a quite variety of application in industries, medicine and science.

→ In the analytical chemistry field most applications like in the asility of ultra sound to extract trace analysis from the solid matrix.

→ Sound waves can induce mechanical vibrations in a solid, liquid and gas

→ where as electromagnetic waves can pass through vacuum, ultrasound has the frequency higher than, the human audible range (1-16 kHz).

→ Sound waves must travel in the matter as they involve expansion and compression, cycles travelling through the medium expansion, pulls molecules apart where as compresion pushes them together

→ In a liquid the expansion cycles produces negative pressure, if ultrasound in strong enough, the expansion cycle can create baboals or cavities in the

liquid, this is so when the negative pressure exterted exceeds the local tensile strength of the liquid, which varies depending upon its nature and purity.

→ The process by which vapour babools form grow and undergo inclosinee collaps is known as cavetation, the whole process taken place about 100 usec

→ A caustic cavification thus provides a unique interaction of energy and matter, the high temprature and pressure produced bead to the formation of free radicals and other compound.

→ Inthis sonification of pure water causes thermal resourciation of water molecule -s into hydrogen atom and 'OH' free radicals, the latter forming H_2O_2 by recombination; the use of an acid extract process.

→ Along the acids employed as extracting HNO_3 is reported to have an anhanced preformance, due to it's oxidation properties

→ There are two common devices for Ultrasound application namely Bath and probe units.

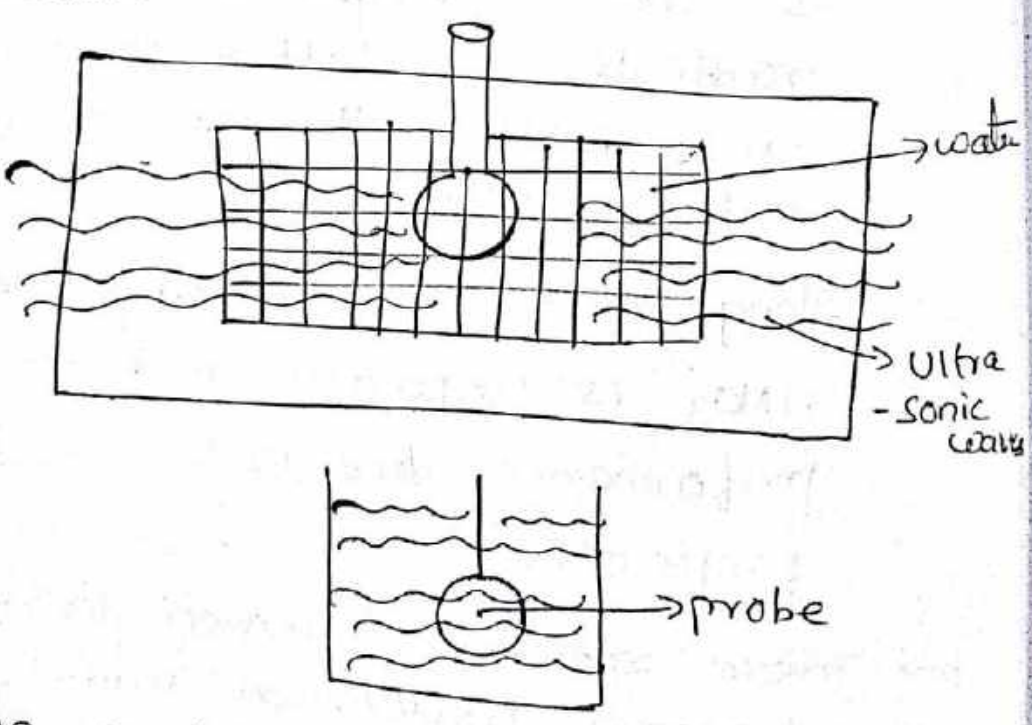
→ Ultrasonic probe have the advantages over ultrasonic bath in that they focus their energy on a localized sample zone.

→ There by providing more efficient cavification in the liquid, the sonification time required with ultrasonic probe in less and that need when using ultrasonic bath.

→ ultrasound assented leaking in an effective way of extracting and facilitating of quick determination of large number of analyses from various environmental samples

→ microwave and ultrasound heatings are wet decomposition techniques.

Ultrasonicator:-



→ using probe we can develop ultrasonic waves then we can decompose the sample.

⇒ Decomposition with water:-

→ magnesium, copper, zinc sulphates also dissolved in water.

→ The portion of calcium carbonate dissolve depends on the contents of CO_2 the distilled water.

→ The no of borates ex:- borate, borax, colemanite and ulexite dissolved in water at elevated temprature.

→ Aq. sodium acchonate soln has been employed for the determination of sulphates.

→ metallic zinc also dissolved and boil with a aq. lead chloride solution

→ decomposition of CaF_2 is carried out by heating with acidified $AlCl_3$ solution on a water bath for one to two hours.

→ Lime stone dissolved in 5% ammonium chloride with continued boiling.

$$NaH + H_2O \longrightarrow NaOH + H_2$$

$$NaBH_4 + 2H_2O \longrightarrow NaBO_2 + 4H_2$$

$$Al_4C_3 + H_2O \longrightarrow 4Al(OH)_3 + 3CH_4$$

$$CaC_2 + H_2O \longrightarrow Ca(OH)_2 + C_2H_2$$

$$Mg_3N_2 + 6H_2O \longrightarrow 3Mg(OH)_2 + 2NH_3$$

$Ca_3P_2 + 6H_2O \longrightarrow 3Ca(OH)_2 + 2PH_3$

$Cl_2O_7 + H_2O \longrightarrow HClO_4$.

$BeC_2 + 4H_2O \longrightarrow 2Be(OH)_2 + CH_4$

$4CaO \cdot Al_2O_3 \cdot Fe_2O_3 + 6H_2O \xrightarrow{\text{hydration}}$

$3CaO\,Al_2O_3\,6H_2O + Fe_2O_3CaO$

colloidal gel

$3CaO\,Al_2O_3 + 6H_2O \xrightarrow{\text{hydrolysis}} 3Ca(OH)_2 + 2Al(OH)_3$

8/10/14

⇒ Fusion:-

Principle:- The fusion mixture is usually fused 10 times of the sample, the fusion mixture and finely powdered sample are mixed the contents in the crucible are heated slowly to remove mixture content and CO_2

→ The crucible is heated to red hot, untill no gas evaluation is visible and the mass is completely fused, the melt is cooled and extracted with H_2O (or) acid.

Ex:- Na_2CO_3 is an excellent fusion material, it's melting point is 850°C, metal silicates are reacted with sodium carbonates, and converted into sodium silicates which are highly soluble.

$$M^+\text{- silicates} + Na_2CO_3 \xrightarrow[30-60\ min]{900-1200^\circ C} M^+CO_3 + Na\text{-silicates}$$

$M^+CO_3 \xrightarrow{H^+ \text{ acid}/H_2O}$ Insolution $M^+ + CO_2\uparrow$

Na-silicates (which are highly soluble)

Reason for Na_2CO_3 selection:-

Sodium is not interfear, and also most of the metal carbonates insoluble we can eliminates carbonates easily from Na_2CO_3.