SUMMARY OF CHEMISTRY PRACTICAL PAPER ADVANCED LEVEL

Question one

Volumetric analysis or Quantitative (titration)

Types of titration

(a)Acid-base titration (using phenolphthalein or methyl orange indicator)

(b) Redox titration

(i) Titration involving potassium permanganate (VII) as the **oxidant**

(ii) Titration involving sodium thiosulphate as the **reductant**

- Iodimetric titration
  Direct titration of iodine and sodium thiosulphate (**adding starch near the end point**)
- Iodometric titration
  Indirect Titration of iodine liberated from the reaction of oxidizing agent such as (MnO_4^-.$Cr_2O_7^{2-}$,IO_3^-,H_2O_2,Cu^{2+}) iodide ions and dilute acid with sodium thiosulphate

Table of results

- Titre values are recorded to 2decimal place.**(e.g ,22.00,34.70)**
- Entries will score if titre values are within the expected range, realistic **(un realistic, include 23.02,54.00)** and there is correct subtraction.
- Titre values to be averaged, must be from **table** and consistent (within ± 0.1)
- Average volume, must be calculated from **two consistent** from the table(within ± 0.5)

Calculations involving

- **Standardizing of solution** (using standard soln e.g,0.2M,1.2g in 250/1000cm^3)
  (i)Calculate moles in a standard solution pipetted or prepared or in reacted volume,(ii)use mole ratio,(iii) calculate moles in 1000cm^3 .
- **Back titration**
  We first add a known volume of standard excess reagent to analyte that reacts completely, after we measure how much was left over (by titration).moles of standard reagent that reacted with analyte = initial moles of standard excess – moles of left over
  Moles of analyte that reacted then determined by using the mole ratio
- **Diluting a more concentrated solution**
  This is when water is added to a known volume of solution up to a fixed volume
  Calculate the moles in dilute solution, Moles after dilution = moles before dilution

- **Analyzing mixtures**
  -Mixture of sodium carbonate and sodium hydroxide by double indicator method
  -Mixture irons (II) and iron (III). $KMnO_4/H^+$ reacts with only iron(II). iron (III) must be reduced first with zinc
  -Mixture of $H_2C_2O_4$ and $Na_2C_2O_4$,NaOH reacts only with $H_2C_2O_4$ while $KMnO_4/H^+$ reacts with both. **Compounds** $FeC_2O_4\ 2H_2O$ and $KH_3(C_2O_4)_2 2H_2O$

Hint

- Read the question several times until you **understand** the **procedure** and aim or **objective** of experiment
- Make a simple **sketch** of apparatus and reagents to help you understand the procedure and to answer questions in logical sequence.
- Calculations are done from **first principles**
- Mind about the **number of decimal places** of your answers(3dps or 3significant figures in standard form)
- Write **logical** and meaningful statements
- Calculate but don't not quote the molar mass of compounds(**use the formula given)**

Rates of reactions and heats reaction

(a) Follow instructions strictly as they have been given
(b)Apparatus commonly used and the number of decimal places used for recording

Apparatus	Quantity	No of dp		No of dp
Thermometer-	Temperature(^oC)	1dp	Reciprocal temperature	
Stop clock	Time(s)	1dp	Reciprocal of time	
Stop watch	Time(s)	1dp or 2dp		

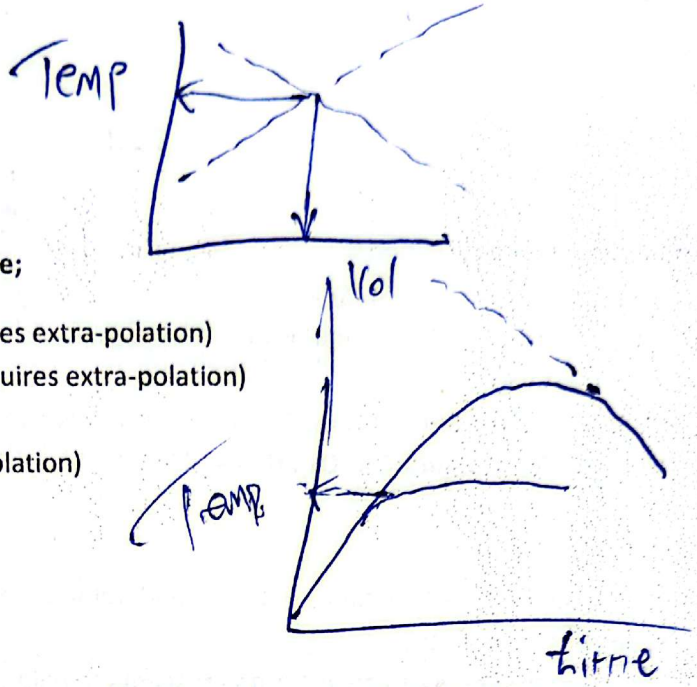
(c) Table of results

Values are expected to follow a certain trend

- Values of temperature increases then decreases
  or Values of temperature increases then levels or remains constant
- For kinetics time or volumes used either increases or decreases

(d)Plotting of graphs

- Appropriate scale should be used(interval of 1,2,5.10,for smaller values use standard for
- The axes must be labeled and with correct units
- Shapes of graphs either straight line or a smooth curve



(e) Use the graphs to determine the;

- Temperature change (requires extra-polation)
- Volume of solution used requires extra-polation)
- Slope,
- Intercept (requires extra-polation)
- Order of reaction

(f) Common Shapes of graphs

Question two

Qualitative analysis in organic; you are required to **analyze** the sample then **identify** the **cations** and **anions** in a given sample

The Sample given may be a compounds or mixer of compounds and will contain **four** ions

Cations are positively charged ions

NH_4^+	**non-transition metal ions** all are white solids when dissolved form colourless solutions	Cu^{2+}	blue/green	**Transition metal ions** solids are colourled and their solutions are also colourled
Ba^{2+},		Cr^{3+}	green/purple	
Ca^{2+},		Fe^{2+},	green	
Mg^{2+}		Fe^{3+}	brown	
Al^{3+},		Ni^{2+}	green	
Pb^{2+},		Mn^{2+}	pale pink/colourless	
Zn^{2+},				
Sn^{2+}				

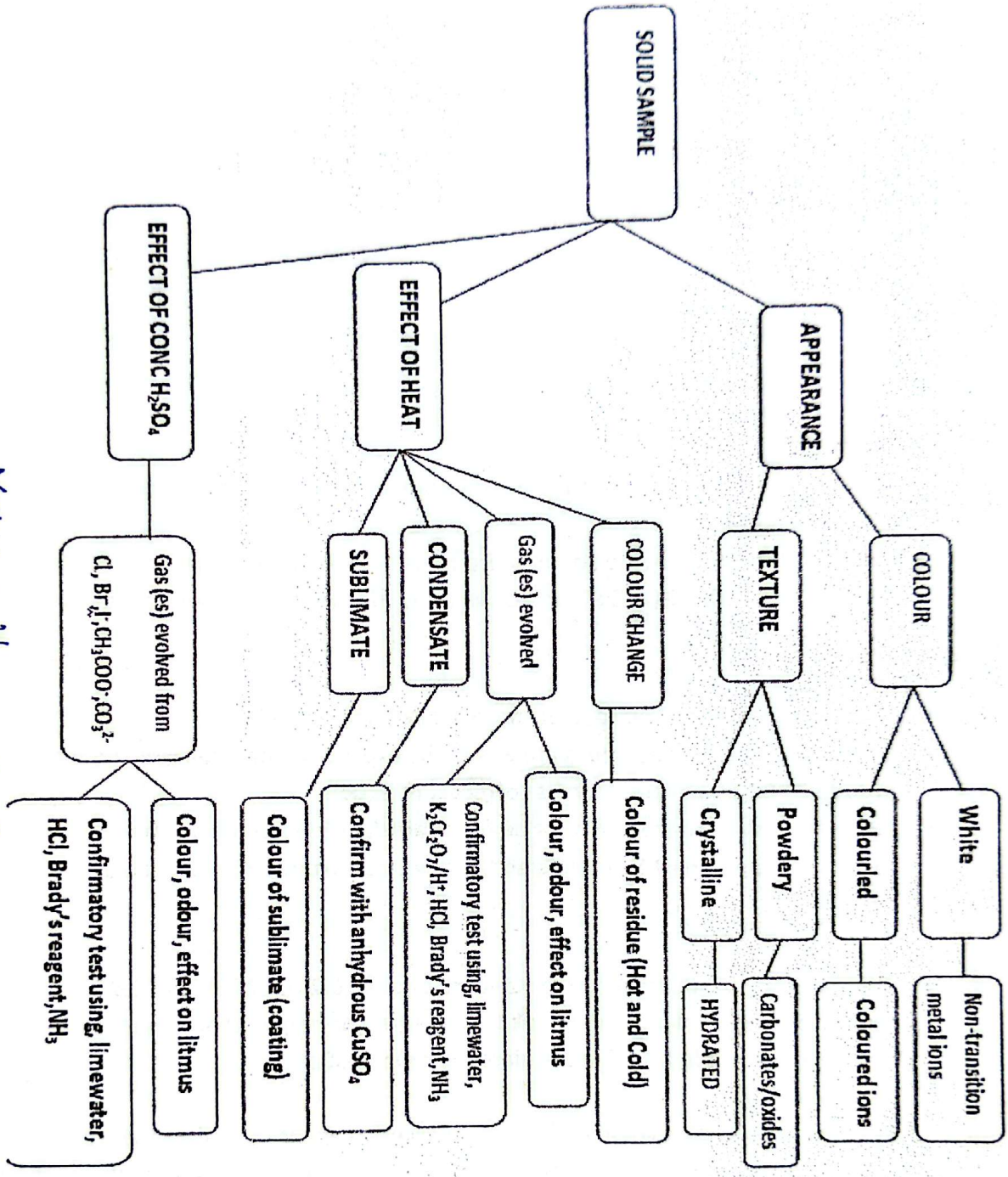
Anions are negatively charged ions; they include

SO_4^{2-} SO_3^{2-} $S_2O_3^{2-}$	$C_2O_4^{2-}$,CO_3^{2-}, CH_3COO^-	Cl^-, Br^- I^-	PO_4^{3-} NO_3^-

Key words, must be correctly used

Precipitate, effervescence, residue, filtrate, dissolves, soluble, insoluble, miscible, evolved, turns, no observable change, partially, correct names of reagents.

PRELIMINARY TESTS ON SOLID SAMPLE

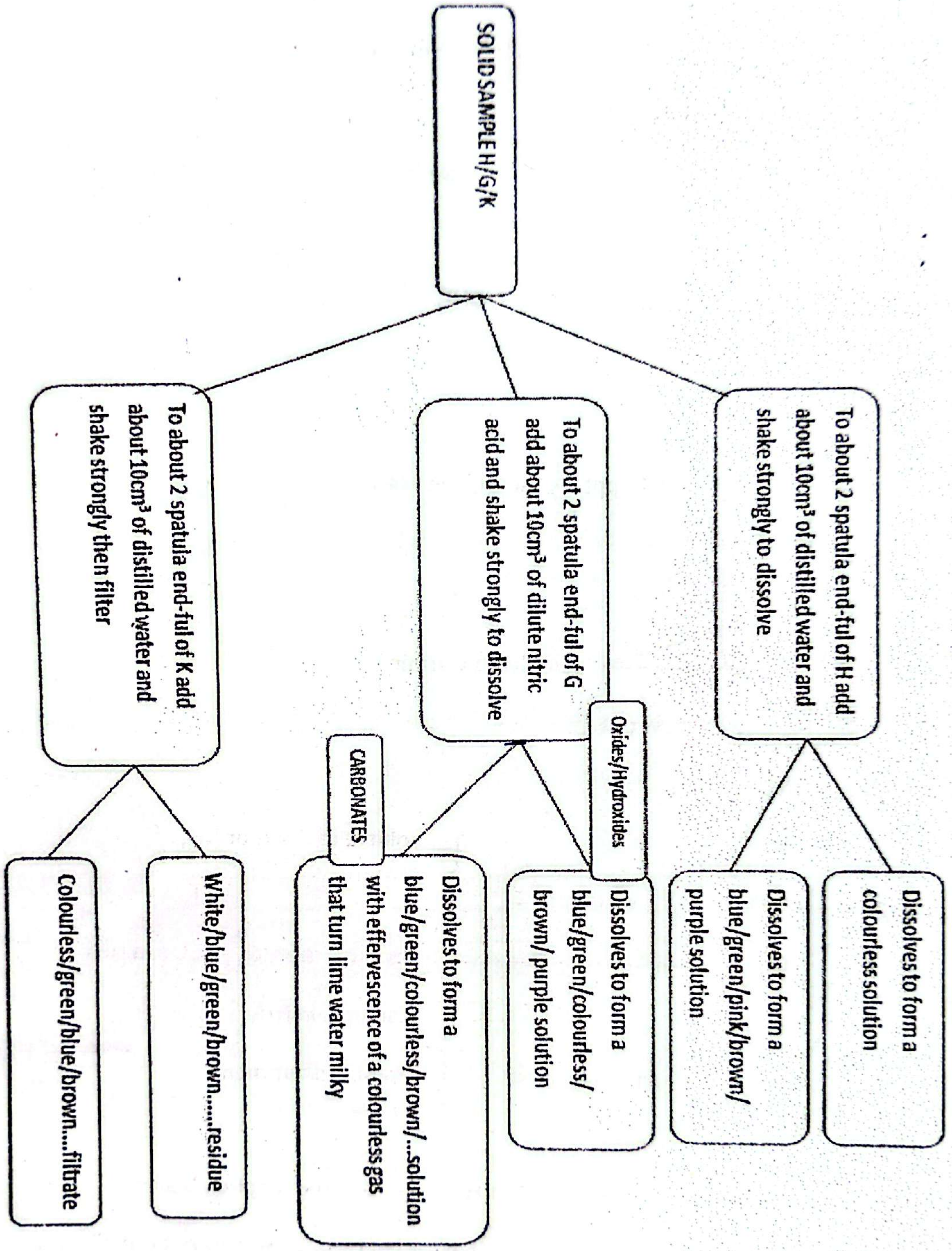


Yellow residue → Anhydrous Ni_2SO_4

$Zn(CH_3COO)_2 \longrightarrow ZnCO_3 + CH_3COCH_3$, $ZnCO_3 \longrightarrow ZnO + CO_2$

White fumes with sweet odour yellow precipitate with 2,4-dinitropheny hydrazine, yellow residue hot white on cooling, black reside, white sublimate,coourless condensate turns anhydrous $CuSO_4$ blue

PREPARING A SOLUTION FOR TEST



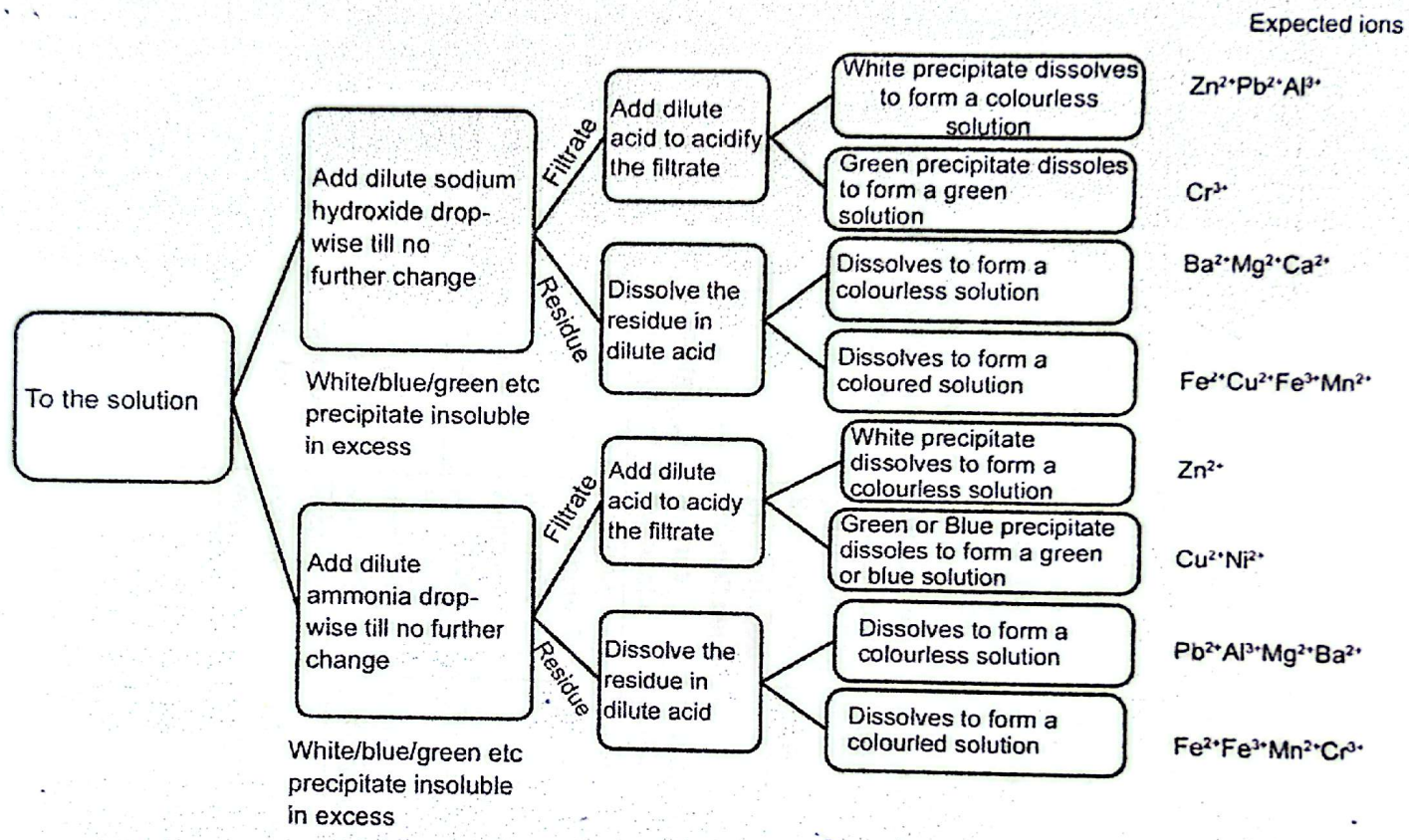
Always write logical deductions from observation made

Always write complete observation, and avoid sweeping statements

Preliminary and confirmatory tests for cations

Cation	Colour	with NaOH	with NH_3	Confirmatory reagent used	Observation
NH_4^+	Colourless	no observation change	no observation change	NaOH then warm	Colourless gas turns litmus blue and
Ca^{2+}		white ppt insoluble in excess	no observation change	$(NH_4)_2C_2O_4$ +CH_3COOH	white ppt insoluble in CH_3COOH
Ba^{2+}			white ppt insoluble in excess	$K_2Cr_2O_4$+NaOH	yellow ppt insoluble in NaOH
Mg^{2+}				NH_4Cl_s +Na_2HPO_4(aq)+NH_3(aq)	white ppt insoluble in excess NH_3
Zn^{2+}		white ppt soluble in excess	white ppt soluble in excess	NH_4Cl_s +Na_2HPO_4(aq)+NH_3(aq	white ppt soluble in excess NH_3
Pb^{2+}			white ppt insoluble in excess	$K_2Cr_2O_4$+NaOH/KI	
Al^{3+}				HCl+ litmus soln+NH_3	blue lake
Cu^{2+}	Blue /green	blue ppt insoluble	blue ppt soluble forming a deep blue solution	$K_4Fe(CN)_6$ KI till soln is blue	-Reddish brown ppt -white ppt in brown soln
Ni^{2+}	Green	green ppt insoluble	**green ppt soluble forming pale blue solution**	NH_3 + Dimethylglyoxime	Pink/red precipitate
Fe^{2+}	Green		green ppt insoluble	$K_3Fe(CN)_6$	dark blue ppt
Cr^{3+}	Green /purple	green ppt soluble to form a green solution	grey green ppt insoluble in excess	NaOH+H_2O_2+ n-Butanol+ H_2SO_4	green ppt soluble,green soln,yellow soln,blue soln
Mn^{2+}	Pale pink /colourless	white ppt ,turn brown insoluble in excess	white ppt ,turn brown insoluble in excess	Conc HNO_3+ solid Sodium bismuthate, Filtrate must be added.	purple or pink solution
Fe^{3+}	Brown	brown ppt insoluble	brown ppt insoluble	$K_4Fe(CN)_6$ or NH_4SCN	Dark blue ppt for Red ppt.

Separation of two cations in aqueous solution using either sodium hydroxide or ammonia



a)use a boiling tube

b)add excess dilute sodium hydroxide or ammonia solution

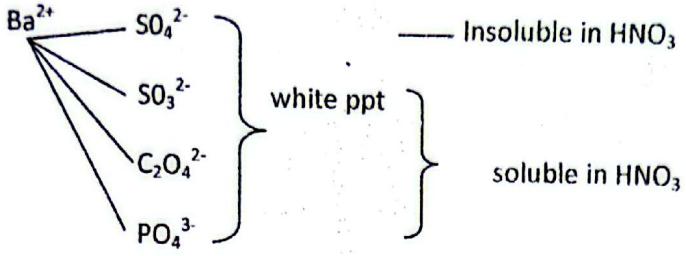
c) to acvidify the filtrate add dilute acid till the precipitate formed just dissolves

d) Pb^{2+}, Ca^{2+}, Ba^{2+} are insoluble in dilute sulphuric acid if used

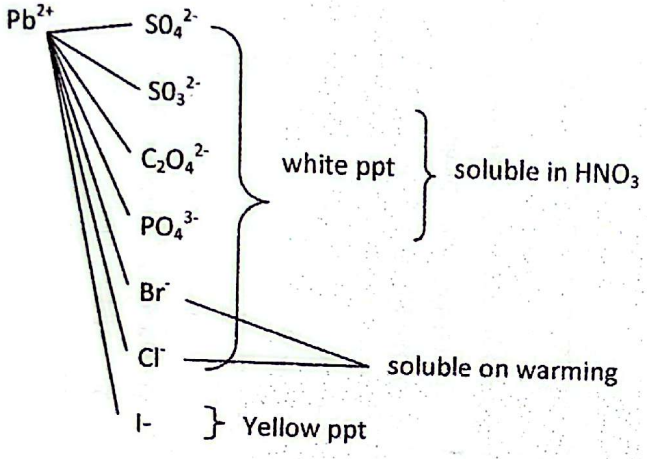
e) Pb^{2+} are insoluble in dilute hydrochloric acid

PRELIMINARY AND CONFIRMATORY TEST FOR COMMON ANIONS.

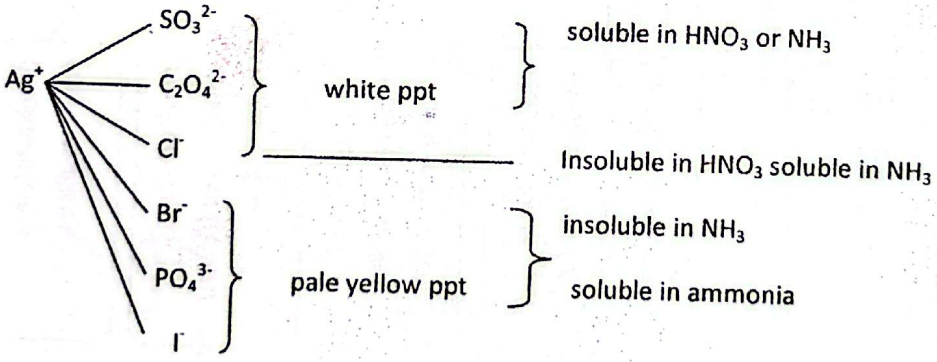
(a)$Ba(NO_3)_2$ + HNO_3 OR $BaCl_2$ +HCl



(b)$Pb(NO_3)_2$ + HNO_3 or warm or $Pb(CH_3COOH)_2$



(c)$AgNO_3$ +HNO_3 or NH_3



(d)CH_3COO^- —— Neutral $FeCl_3$ + Heat —— Brown ppt on boiling

Ethanol + conc H_2SO_4 + heat —— Sweet fruity smell

(e) NO_3^- —— copper turning + conc H_2SO_4 + heat ------- pale brown fumes, pungent, blue litmus red

Zinc powder + NaOH + warm.....Colourless gas turns moist red paper litmus blue

Anion	$K_2Cr_2O_7/H^+$	$KMnO_4/H^+$	I_2	dil HCl + warm
SO_3^{2-}	Orange solution turns green	Purple solution turns colourless	Brown solution turns colourless	effervescence of a colourless gas turns $K_2Cr_2O_7/H^+$ green
$C_2O_4^{2-}$	No observable change	Purple solution turns colourless on warming	No observable change	No observable change

Anion	H_2O_2/H^+	HNO_3 + Bleaching powder + $CHCl_3$ Shake and allow to stand
Br^-	reddish brown solution	Orange solution in organic layer
I^-	brown solution	Purple solution in organic layer

PO_4^{3-} — Conc HNO_3 + Ammonium molybdate + NH_3 ——— Yellow ppt soluble in NH_3

PO_4^{3-} — $FeCl_2$ ——— yellow ppt

Soluble CO_3^{2-} — $Ba(NO_3)_2 + HNO_3$ } white ppt dissolves in acid with effervescence of a colourless gas

Soluble CO_3^{2-} — $Pb(NO_3)_2 + HNO_3$ } white ppt dissolves in acid with effervescence of a colourless gas

Soluble CO_3^{2-} — $AgNO_3 + HNO_3$ } white ppt dissolves in acid with effervescence of a colourless gas

Anions	$MgSO_4$
CO_3^{2-}	white precipitate
HCO_3^-	mo observable change in cold, white ppt on heating

() concentrated sulphuric acid and warm

conc H_2SO_4 —
- NO_3^- -------Pale brown fumes
- Cl^- .....white fumes ,forms dense white fumes with conc HCl
- Br^- ..... reddish brown fumes
- I^- .....Purple vapour
- CH_3COO^- ..... White fumes with vinegar smell
- CO_3^{2-} ....effervescence of colourless gas turns lime water milky

Question three

Qualitative analysis organic;

You are required to **analyze** and determine the **nature** of organic compound

Nature of organic compound;

(i)Structure of organic compound	(ii) Functional group	(iii) classification of functional group
Aliphatic or Aromatic	Hydroxyl, Carbonyl, Carboxyl etc	Primary, secondary ,tertiary for alcohols Ketones or Aldehydes for carbonyl Position of hydroxyl group or carbonyl group on primary/secondary or aldehyde/ketones

Comment on the nature

Aliphatic primary alcohol with the structure

Aromatic primary alcohol

Secondary alcohol with carboxyl group

Aromatic unsaturated carboxylic acid

Aliphatic carboxylic acid with a chloro group

Preliminary tests

Test	Expected observations	Expected deductions
Combustion or flame test	**white crystalline solid or colourless liquid** burns with blue or yellow non-sooty flame burns with yellow sooty flame	**Aliphatic** compound with **low carbon** content **Aromatic** compound with **high carbon** content
Solubility in water	**liquid organic compound** -miscible in water forming colourless soln -partially miscible **solid organic compound** -dissolves forming colourless soln -sparingly soluble dissolves on heating	**Polar** aliphatic of low molecular mass **Polar** aromatic/polar aliphatic of high molecular mass polar aliphatic of low molecular mass polar aromatic/polar aliphatic of high molecular mass
Solubility in NaOH	Dissolves to form a colourless solution	Acidic compound (carboxylic or phenol)
Effect of aqueous solution on litmus paper	No effect on **both** blue and red litmus papers turns blue litmus paper red turns red litmus paper blue	Neutral compound acidic compound basic compound

Ca^{2+}
Mg^{2+}
Ba^{2+}
} White ppt insoluble in excess NH_3

Chemical tests; identification of functional group

Reagents used to confirm the presence or absence of a particular functional group

reagents	expected observation	expected deductions
$NaHCO_3/Na_2CO_3$/Mg powder	effervescence of colourless gas	carboxylic acid present
Brady's reagent	yellow or orange ppt	carbonyl compounds present
Alcohol+ conc H_2SO_4 +Heat	sweet fruity smell	carboxylic acid present
Carboxylic acid + conc H_2SO_4 +Heat	sweet fruity smell	alcohol present
$FeCl_3$	brown ppt	benzoic acid present
Neutral $FeCl_3$	purple colouration	phenol present
Hot $NaOH+HNO_3+AgNO_3$	white/cream/pale yellow ppt	aliphatic compound with chloro/Bromo/Iodo group

N.B If the expected observation has not occurred we say no observable change

The flow of the test in the experiment is crucial as you are making deductions

Tests for reducing agents: Tests more in saturation in the crd.

Chemical tests; Reagents used to classify a particular functional group

$KMnO_4/H^+$ +heat or warm	purple solution turns colourless	Reducing agents 1° & 2° alcohols Aldehydes Methanoic acid H–C(=O)–OH
$K_2Cr_2O_7/H^+$ +heat or warm	orange solution turns green	
luca's reagent (anhydrous $ZnCl_2$+Conc HCl)	-immediate cloudy solution	Tertiary
	cloudy soln in 6th 7th 8th 9th or 10th minute	Secondary
	-no observable change	Primary.
Ammoniacal silver nitrate ($AgNO_3$+NaOH+NH_3) warm	Silver mirror	Aldehyde or Methanoic
Fehling solution & heat	Reddish brown precipitate	
Saturated Sodium hydrogen sulphite solution	white precipitate/colourless crystals	$CH_3C(R)(OH)SO_3Na$ or Aldehyde (methyl ketones)
Iodine + NaOH	yellow precipitate	$CH_3CH(OH)$ $CH_3C(=O)$

Primary CH_3CH_2OH alcohol / 2° alcohol with structure $CH_3CH(OH)$

Aldehyde CH_3CHO or Ketone with structure $CH_3C(=O)$

16000
110cc 780

Challenges

Logical deductions from observations made from tests on products formed

TESTS	OBSERVATION	DEDUCTION
(a)add water and test with litmus	miscible to form colourless solution, no effect on litmus papers	Neutral compound eg alcohol, carbonyl cmd
(b) To 1cm^3 of Q add little Brady's reagent	no observable change	Carbonyl compound absent.
(c) To 1cm^3 of Q add $K_2C2r_2O_7/H^+$ add heat divide into two	orange solution turns green	Primary, 2° alcohol
(c)(i)To 1st portion add Brady's reagent	yellow precipitate	Primary alcohols oxidised to aldehydes/ Secondary oxidised to ketones.
(c)(i)To 2nd portion add Fehling solution	no observation change	Secondary alcohol to ketone. was oxidised
(d)To 1cm^3 of Q add 1cm^3 of ethanoic acid plus 4drops of conc H_2SO_4 and heat	sweet fruity smell	Esterification rxn, Ester formed Secondary alcohol present.
(e) To 1cm^3 of Q add conc sulphuric acid and heat pass the vapour in $KMnO_4/H^+$	white fumes turns $KMnO_4/H^+$ colourless	Secondary alcohol dehydrated to form an alkene.

-Logical deduction will depend on the flow of the tests in an experiment

TESTS	OBSERVATION	DEDUCTION
(a) To 1cm^3 of Q add little Brady's reagent	yellow precipitate	Carbonyl present.
(b) To 1cm^3 of Q add $K_2C2r_2O_7/H^+$ add heat	orange solution turns green	Aldehyde
or		
(a) To 1cm^3 of Q add $K_2C2r_2O_7/H^+$ add heat	orange solution turns green	1° or 2° alcohol aldehyde, Methanoic
(b) To 1cm^3 of Q add little Brady's reagent	yellow precipitate	Aldehyde