



Teachers' Version w/o Prep list

CATHOLIC JUNIOR COLLEGE

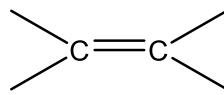
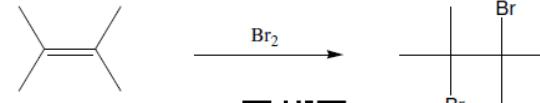
H2 CHEMISTRY 9729

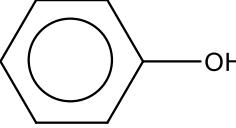
2020 JC2 PRACTICAL HANDBOOK – Book 4

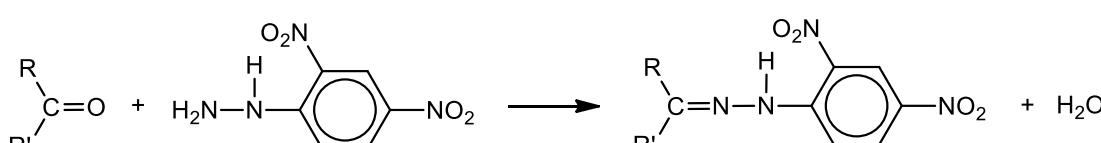
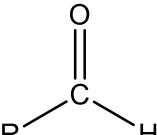
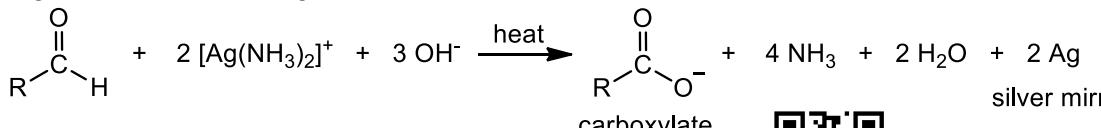
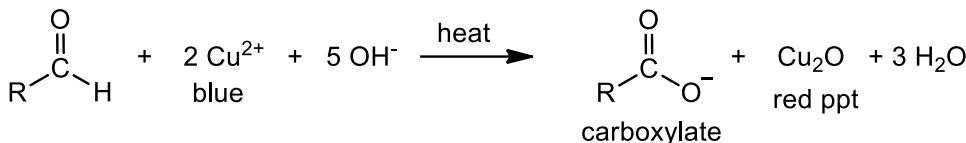
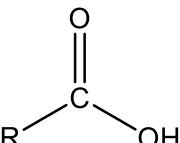
7.1 Organic Qualitative Analysis and Synthesis

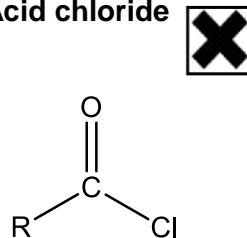
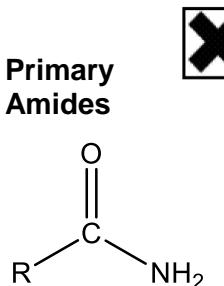
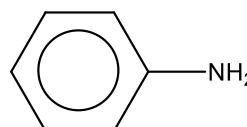
7.1.1 Organic Qualitative Analysis - Identification of Functional Groups

Simple chemical tests to identify functional groups in organic compounds:

Test for	Procedures	Observations
Alkenes  	<ol style="list-style-type: none"> Add 1-2 drops of Br₂(aq) to 1 cm³ of organic compound. Shake well after adding each drop of Br₂(aq). [electrophilic addition of alkenes] - test for unsaturation. Add 1-2 drops of acidified KMnO₄(aq) to 1 cm³ of organic compound. Heat in water bath for 2-3 mins. [oxidation of alkenes] 	<p>1. <u>Orange</u> Br₂(aq) rapidly <u>decolourised</u>. [Br₂ adds across C=C bond of alkenes.]</p> <p>2. <u>Purple</u> acidified KMnO₄(aq) <u>decolourised</u>.</p> <p>Note: Methylbenzene, aldehydes and alcohols (1°, 2°) also give +ve test with hot acidified KMnO₄</p> <p>Relevant video of chemical test: https://tinyurl.com/y7jftf2a</p>  
Halogenoalkanes R-X where R = alkyl, X = Cl, Br, I	<ol style="list-style-type: none"> Add 1 cm³ of NaOH(aq) to 1 cm³ of organic compound. Heat in water bath for 5 mins. Then add 1 cm³ of dilute HNO₃, followed by 2-3 drops of AgNO₃(aq). [hydrolysis, followed by precipitation] 	<p>1. with R-Cl - <u>white ppt</u> of AgCl(s) observed; with R-Br - <u>cream ppt</u> of AgBr(s) observed; with R-I - <u>yellow ppt</u> of AgI (s) observed.</p> <p>[Dilute nitric acid is added to prevent precipitation of Ag₂O (brown ppt).]</p> <p>Relevant video of chemical test: https://tinyurl.com/test-for-halogenoalkanes (watch till 1:29 min)</p> 
Alcohols  1° alcohol, RCH ₂ OH 2° alcohol, R ₂ CHOH 3° alcohol, R ₃ COH	<ol style="list-style-type: none"> Add 2-3 drops of acidified KMnO₄(aq) to 1 cm³ of organic compound. Heat in water bath for 2-3 mins. [oxidation of alcohols] 	<p>1. with 1° alcohol - purple KMnO₄(aq) decolourises; <i>carboxylic acid formed</i> with 2° alcohol - purple KMnO₄(aq) decolourises; <i>ketone formed</i>. with 3° alcohol - solution remains purple (<i>no reaction</i>). [Purple MnO₄⁻ reduced to colourless Mn²⁺.]</p> <p>Note: Aldehydes also give +ve test with hot acidified KMnO₄.</p> <p>Video of oxidation of ethanol: https://goo.gl/ta8aqS</p> 

Test for	Procedures	Observations
Alcohols  1° alcohol, RCH ₂ OH 2° alcohol, R ₂ CHOH 3° alcohol, R ₃ COH	<ol style="list-style-type: none"> Add 2-3 drops of acidified K₂Cr₂O₇(aq) to 1 cm³ of organic compound. Heat in water bath for 2-3 mins. [oxidation of alcohols] Add 1 cm³ of ethanoic acid to 1 cm³ of organic compound, followed by a few drops of conc. H₂SO₄. Heat in water bath for 5 mins and then pour into water. [esterification] 	<ol style="list-style-type: none"> with 1° alcohol - orange K₂Cr₂O₇(aq) turns green; carboxylic acid formed with 2° alcohol - orange K₂Cr₂O₇(aq) turns green; ketone formed. with 3° alcohol - solution remains orange (<i>no reaction</i>). [Orange Cr₂O₇²⁻ reduced to green Cr³⁺.] <p>Note: Aldehydes also give +ve test with hot acidified K₂Cr₂O₇.</p> <p>Relevant video of chemical test: https://tinyurl.com/dichromateinalcohol</p> 
Phenols  	<ol style="list-style-type: none"> Add 2-3 drops of neutral FeCl₃(aq) to 1 cm³ of organic compound. [complex formation] To 1 cm³ of organic compound, add Br₂(aq) dropwise until in excess. Shake well after adding each drop of Br₂(aq). [electrophilic substitution] 	<ol style="list-style-type: none"> Violet coloration observed. <p>Relevant video of chemical test: https://tinyurl.com/testforphenol (watch till 2:00 min)</p>  $\text{C}_6\text{H}_5\text{OH} + 3 \text{Br}_2(\text{aq}) \rightarrow \text{C}_6\text{H}_3(\text{Br})_3\text{OH} + 3 \text{HBr}$ <p>Note: Phenylamine also gives the same observation.</p> <p>Relevant video of chemical test: https://tinyurl.com/bromineinphenol</p> 

Test for	Procedures	Observations
Carbonyl compounds (Aldehydes & Ketones) 	1. Add 2 cm ³ of 2,4-dinitrophenylhydrazine reagent to 1 cm ³ of organic compound. If precipitate is not formed immediately, warm in water bath. <i>[condensation reaction]</i>	1. <u>Orange ppt</u> formed.  Relevant video of chemical test: https://tinyurl.com/y8f4rhtt 
Aldehydes 	1. Add 1 cm ³ of Tollens' reagent (ammoniacal silver nitrate) to 1 cm ³ of organic compound. Warm in water bath for 5 mins. <i>[oxidation of aldehydes]</i>	1. <u>Silver mirror</u> formed on the side of the test tube. <i>[Ag⁺ ions reduced to Ag.]</i>  Relevant video of chemical test: https://goo.gl/WNKs85 
	2. Add 1 cm ³ of Fehling's solution to 1 cm ³ of organic compound. Warm in water bath for 5 mins. <i>[oxidation of aldehydes; <u>not</u> for benzaldehyde]</i>	3. <u>Brick red ppt</u> of Cu ₂ O formed.  Relevant video of chemical test: http://tinyurl.com/Fehling-solution
Carboxylic acids 	1. Add a small spatula of NaHCO₃(s) or Na₂CO₃(s) to 1 cm ³ of organic compound. 2. Bubble the gas evolved into limewater. <i>[salt formation]</i>	1. <u>Brisk effervescence</u> ; colourless gas gives <u>white precipitate</u> with limewater. CO ₂ (g) evolved. $\text{RCOOH} + \text{NaHCO}_3 \rightarrow \text{RCO}_2^- \text{Na}^+ + \text{H}_2\text{O} + \text{CO}_2 \text{ (effervescence)}$ 

Test for	Procedures	Observations
Acid chloride 	<ol style="list-style-type: none"> Add 1 cm³ of AgNO₃(aq) to 1 cm³ of organic compound. [hydrolysis, followed by precipitation] Add 1 cm³ of ethanol to 1 cm³ of organic compound. Pour mixture into water. [esterification] 	<ol style="list-style-type: none"> <u>White ppt of AgCl formed.</u> $\text{RCOCl} + \text{AgNO}_3 \rightarrow \text{RCO}_2\text{H} + \text{AgCl} + \text{HNO}_3$ A 'pleasant' characteristic smell of ester detected. $\text{CH}_3\text{CH}_2\text{OH} + \text{RCOCl} \rightarrow \text{RCO}_2\text{CH}_2\text{CH}_3 + \text{HCl}$ <u>Note:</u> This is not a preferred diagnostic test for alcohols the 'ester smell' may be subjective.
Primary Amides 	<ol style="list-style-type: none"> Add ½ spatula of organic compound to 5 cm³ of NaOH(aq). Heat in water bath for 5 mins. Test the gas evolved with moist red litmus paper. Dip a glass rod in conc. HCl and bring it close to the gas evolved. [hydrolysis of amide] 	<ol style="list-style-type: none"> Pungent, colourless gas turns moist red litmus paper <u>blue</u> and gives <u>dense white fumes</u> (of NH₃(g)) with conc HCl. NH₃(g) evolved. $\text{R}-\overset{\text{O}}{\underset{\text{C}}{\text{ }}}-\text{NH}_2 \xrightarrow[\text{warm}]{\text{NaOH(aq)}} \text{R}-\overset{\text{O}}{\underset{\text{C}}{\text{ }}}-\text{O}^- + \text{NH}_3$
Phenylamine 	<ol style="list-style-type: none"> To 1 cm³ of organic compound, add Br₂(aq) dropwise until in excess. Shake well after adding each drop of Br₂(aq). [electrophilic substitution] 	<ol style="list-style-type: none"> <u>Orange Br₂(aq) decolourised;</u> <u>white ppt formed, and steamy fumes</u> of HBr. $\text{C}_6\text{H}_5\text{NH}_2 \xrightarrow{\text{Br}_2(\text{aq})} \text{Br-C}_6\text{H}_3\text{NH}_2 + 3\text{HBr}$ <p style="text-align: center;">White ppt</p> <p><u>Note:</u> Phenol also gives the same observation.</p>

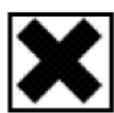
Test for	Procedures	Observations
Esters 	<ol style="list-style-type: none"> Add 1 cm³ of NaOH(aq) to 1 cm³ of organic compound. Heat in water bath for about 3 mins. Then add 2-3 drops of acidified K₂Cr₂O_{7(aq)} to the resulting mixture. Heat in water bath for 2-3 mins. 	<ol style="list-style-type: none"> Orange K₂Cr₂O_{7(aq)} turns green. [Orange Cr₂O₇²⁻ reduced to green Cr³⁺ only if the alcohol is primary or secondary.] [Hydrolysis of ester to give carboxylic acid and alcohol; followed by oxidation of alcohol.]
Iodoform test for and alcohols with CH ₃ CH(OH)- group	<ol style="list-style-type: none"> Add 1 cm³ of NaOH(aq) to 1 cm³ of organic compound. Then add I_{2(aq)} dropwise until the colour of iodine persists. Shake well after adding each drop of I_{2(aq)}. Heat in water bath for about 2 mins, and then add NaOH(aq) dropwise to discharge the excess iodine. Cool the mixture. 	<ol style="list-style-type: none"> Yellow ppt of CHI₃ formed. $\text{R}-\text{C}(=\text{O})-\text{CH}_3 + 3 \text{I}_2 + 4 \text{NaOH} \longrightarrow \text{R}-\text{C}(=\text{O})-\text{O}^-\text{Na}^+ + 3 \text{NaI} + 3 \text{H}_2\text{O} + \text{CHI}_3 \text{ (s)}$ <p style="color: blue;">Relevant video of iodoform test: https://goo.gl/kAxGIQ</p> 



Flammable

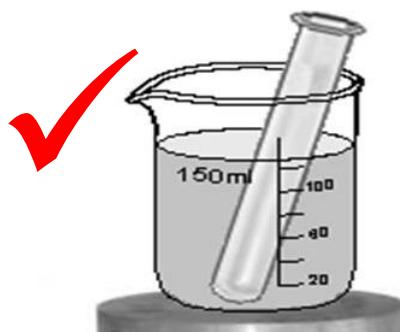


Corrosive

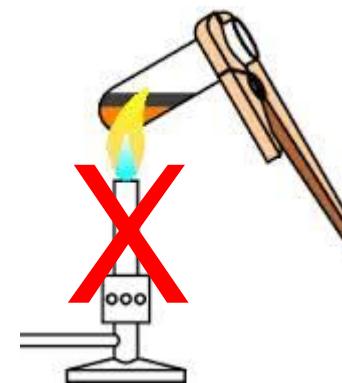


Harmful / Irritant

- (1) Short chain organic compounds which are liquid at room temperature are usually volatile. They should be capped in bottles when not in use. Clean up any spillage immediately to prevent spread of volatile vapour.
- (2) Organic compounds are highly flammable. If heating is required, use a water bath instead of direct heating using a naked flame.



Heating using hot water bath



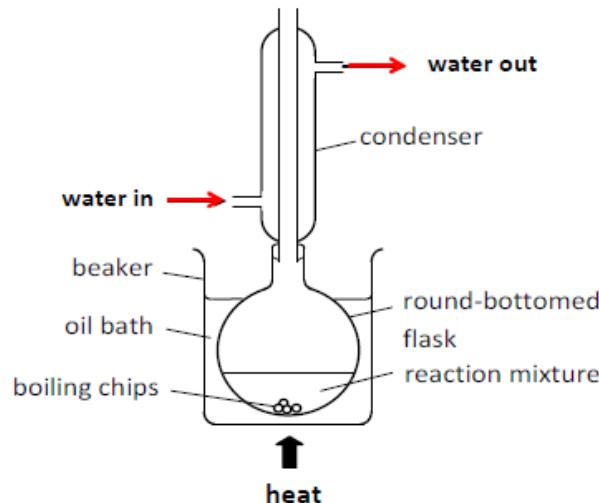
Do NOT heat using a naked flame.

- (3) If acidic fumes e.g. HCl or HBr are liberated, perform the experiment in the fume cupboard and wear goggles to protect eyes from the fumes.
- (4) If alkaline gas is liberated, wear goggles to protect eyes from the fumes. Take care not to bring the mouth of test tube too close to your nose.
- (5) Take care when handling corrosive chemicals; e.g. concentrated acids. Wear gloves so as to avoid direct contact.
- (6) Take care when handling chemicals that are “harmful/irritant” as they may cause reddening or blistering of the skin. Avoid direct contact.
- (7) After completing each chemical test, dispose of the chemicals in proper waste containers (usually placed in fume cupboard). **Do not flush them down the sink, as some organic compounds are toxic/corrosive.**

7.1.2 Organic Synthesis

Set up used for organic synthesis

1. Heating under reflux



Many organic reactions are quite slow and require heating to achieve a reasonable reaction rate. Most organic chemicals are quite volatile, and they will evaporate and be lost when heated.

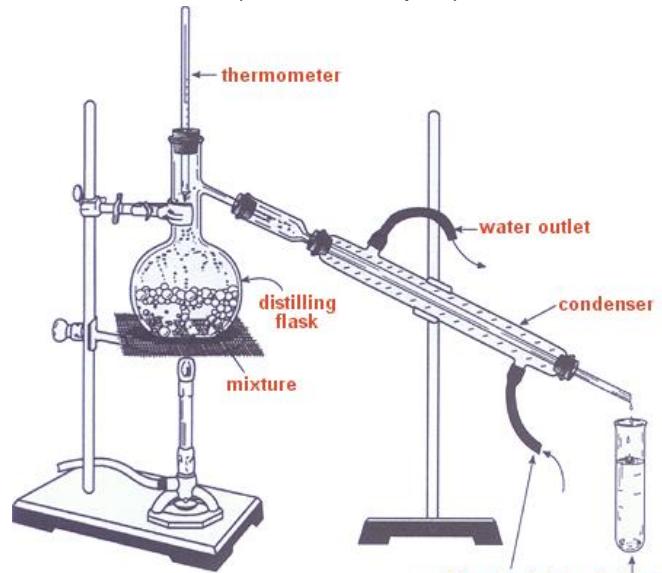
Heating under reflux allows the vapour to cool down at the surface of the condenser, condense back to liquid state and drip back into the flask.

<https://youtu.be/b6xFAEkjmgG>



Methods used for purification:

1. Distillation(to collect liquid)



Distillation separates the component substances from a liquid mixture as different liquids boil off at different temperatures.

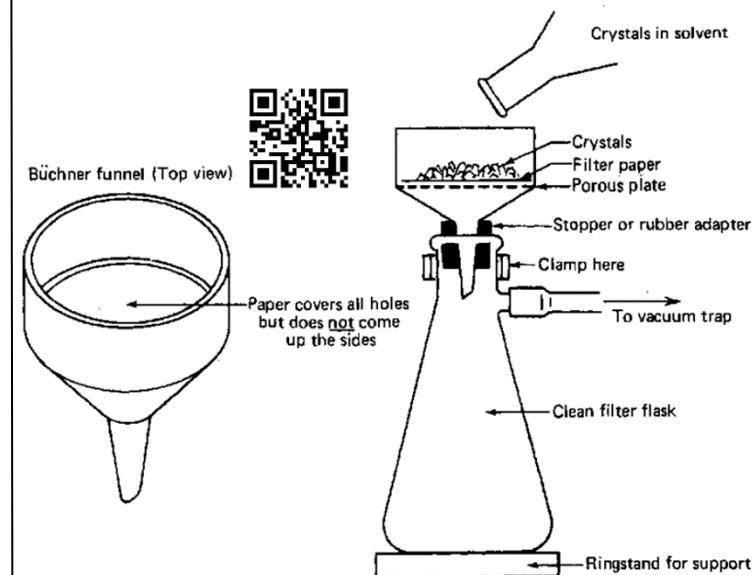
The distillate is then collected once the vapour condenses.

<https://youtu.be/7g4e3dhtgil?t=277>



Methods used for purification:

2. Recrystallisation (to collect solids)



Steps for recrystallisation:

1. Dissolve solids in minimum amount of hot solvent.
2. Filter the solution quickly while hot.
3. Cool hot filtrate (in ice bath).
4. Collect crystals using vacuum filtrate (Büchner funnel).
5. Dry crystals between layers of filter paper/ under infra-red la

* To check for purity: Pure solids melt sharply over a small range of temperature *

7.1.3 Separation of two immiscible liquids

In order to separate immiscible liquids (liquids that do not dissolve in each other), a separating funnel is used. This technique is useful in separating organic products from an aqueous reaction mixture. This technique is also useful in determining the partition coefficient, K , of an organic compound in water. Partition Coefficient, $K = \frac{\text{conc of Organic compound in aqueous layer}}{\text{conc of Organic compound in organic layer}}$

Watch this video to find out how to use a separating funnel
<https://youtu.be/BQMzcfw8w6w>

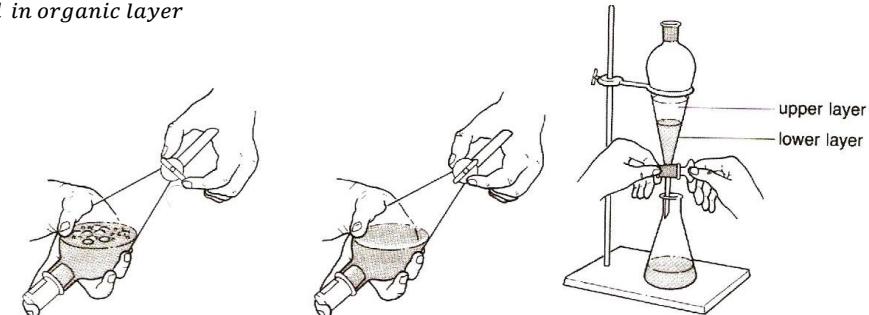
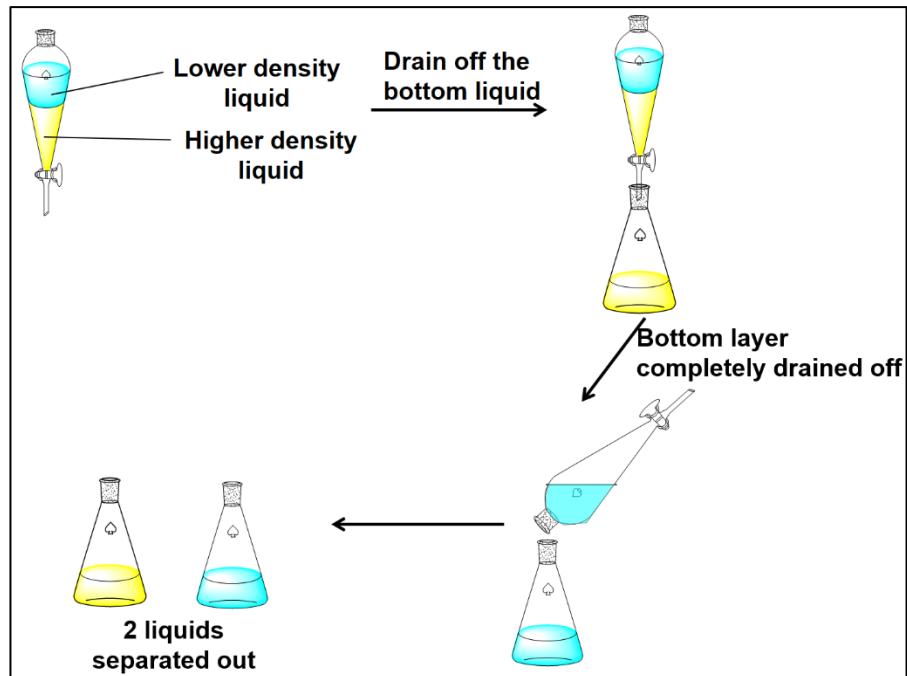


Diagram from http://wwwchem.uwimona.edu.jm/lab_manuals/c1901exp8.html

Procedures

- Pour the two immiscible liquids into the separating funnel. Shake the mixture in the separating funnel and ensure that the pressure in the funnel should be periodically released.
- Support the separating funnel using a retort stand. Then, please a clean conical flask below the separating funnel.
- Allow it to stand and wait for the liquids to separate completely. The denser liquid will be the bottom layer.
- Open the tap of the funnel to allow the bottom layer to drain into the conical flask. Close the tap before the top layer of liquid runs out.
- Invert the separating funnel to allow the top layer of liquid to be collected in another clean conical flask.



7.1.4 Examples of planning Organic Chemistry experiments

Organic Chemistry experiments at A level can be classified as two types,

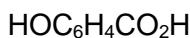
1) Synthesis of Organic Compounds and 2) Distinguishing tests to identify unknown.

Example 1: Synthesis of Organic Compound (2011 A level P2Q1)

{guides, clues and proposed answers are in **bold** and in boxes}

Aspirin was one of the first drugs to be produced synthetically and is one of the most widely used 'over-the-counter' drugs in the world. It is a white solid and is prepared by an acylation reaction between 2-hydroxybenzenecarboxylic acid and ethanoic anhydride.

The other product of this reaction is ethanoic acid.



2-hydroxybenzenecarboxylic acid



ethanoic anhydride



aspirin

2-Hydroxybenzenecarboxylic acid is a white crystalline solid.

Ethanoic anhydride is a corrosive liquid which has a density of 1.08 g cm^{-3} .

Pure aspirin has a melting point of 135°C .

Take note of the given states and information.

Like most organic reactions, the yield of this reaction is less than 100%. Using the procedure described below, a typical yield of pure aspirin is 75%.

Equimolar amounts of 2-hydroxybenzenecarboxylic acid and ethanoic anhydride are used, together with about 8–10 drops of 85% phosphoric acid which catalyses the reaction. When mixing the reactants, the initial reaction may be violent.

The reaction mixture is then heated under reflux conditions for around fifteen minutes. About $2\text{--}3 \text{ cm}^3$ of water is then added to the hot reaction mixture to hydrolyse any unreacted ethanoic anhydride. The addition of this water may cause the mixture to boil.

Once the reaction has subsided, the reaction mixture is poured into about 50cm^3 of cold water. This causes the aspirin to precipitate. The crude aspirin product is purified by recrystallisation from water.

(a) Using the information given above:

[3]

(i) write a balanced equation for the formation of aspirin;



[Note: No H_2O is formed as the product, read question.]

(ii) calculate the masses of reactants you would use to prepare 10 g of pure aspirin, showing your working.

M_r of aspirin = 180.0, M_r of $\text{HOC}_6\text{H}_4\text{CO}_2\text{H}$ = 138.0, M_r of $(\text{CH}_3\text{CO})_2\text{O}$ = 102.0

Amount of aspirin needed = $\frac{10}{180.0} = 0.05556 \text{ mol}$

Since reaction is 75% yield, amount of reactants to use = $\frac{100}{75} \times 0.05556 = 0.07407 \text{ mol}$

[Note: do not forget reaction only gives 75% yield]

Mass of $\text{HOC}_6\text{H}_4\text{CO}_2\text{H}$ to use = $0.07407 \times 138.0 = 10.2 \text{ g}$

Mass of $(\text{CH}_3\text{CO})_2\text{O}$ to use = $0.07407 \times 102.0 = 7.56 \text{ g}$

(b) Write a plan for the preparation of 10g of pure aspirin.

[8]

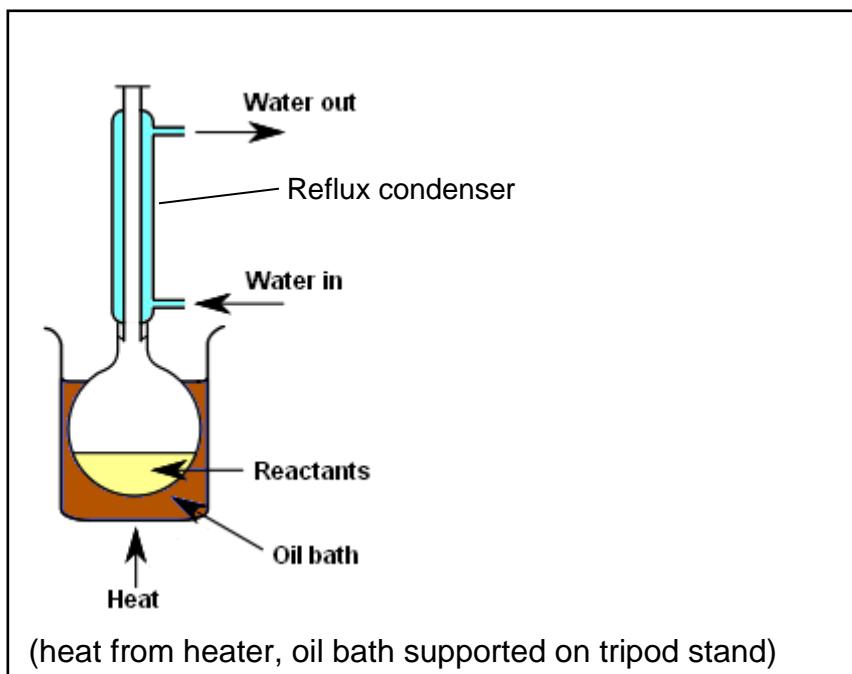
In your plan you should:

draw a diagram of the assembled apparatus you would use when heating the reaction mixture;

give a full description of the procedures you would use to prepare and purify the aspirin;

explain how you would check the purity of your sample.

[Note: No credit merely by rewriting or rephrasing the outline instructions given in question. Give further details of method and explain how each step would be carried out with due attention to obtaining the required product in a pure state by a safe method.]



[Note: Need to show round-bottom flask with a Liebig condenser. Water has to enter and leave condenser in correct manner. The top of condenser should not be closed with a bung or a thermometer. The drawing should not be for heating under distillation, or worse, for fractional distillation. And just heating reactants in a beaker and conical flask using a Bunsen flame shows no understanding of organic reactions that need to be carried out under reflux conditions.]

[Unnecessary diagrams like measuring cylinder and beaker on balance need not be given.]

Procedure

1. Cool about 50 cm^3 and 20 cm^3 of water respectively in two 400 cm^3 beakers using an ice/water bath.
2. Weigh out 10.22 g of 2-hydroxybenzenecarboxylic acid using an electronic mass balance on dry weighing paper. Carefully transfer to a 250 cm^3 round bottomed flask.
3. Carefully measure out 7 cm^3 of ethanoic anhydride using a 10 cm^3 graduated cylinder and transfer to the flask. Swirl the flask to wet the 2-hydroxybenzenecarboxylic crystals. Calculation of volume of ethanoic anhydride used = $7.556 \div 1.08 = 6.996 = 7.0\text{ cm}^3$ [Note: A number of students suggested weighing ethanoic anhydride directly on a balance without appreciating that the question mentions it is a corrosive liquid. Use density data to calculate out volume to be measured.]
4. Using a dropper, carefully add 8-10 drops of 85% phosphoric acid, to the above mixture, spanning over a few minutes to control the rate of reaction. [Note: This precaution of adding conc. H_3PO_4 must be mentioned, many students just rewrote what is listed in question.]
5. Rearrange the apparatus as shown above for heating under reflux. Gently heat the flask in a boiling water/oil bath for about 15 minutes.

6. Remove the flask from the hot water/oil bath and add $2-3\text{ cm}^3$ of ice water using another 10 cm^3 graduated cylinder to decompose any excess ethanoic anhydride. [Note: How the small amount of water is to be added has to be mentioned explicitly so as to be safe, many students just rewrote what is listed in question.]
7. Pour the contents of the flask into the 50 cm^3 of cold water prepared in step 1. Continue to immerse the beaker in the ice/water bath until crystals of aspirin no longer form, stirring occasionally to decompose residual ethanoic anhydride.
8. Set up a vacuum filtration apparatus. Wet the filter paper in the Buchner funnel with $1-2\text{ cm}^3$ of distilled water. Turn on the water aspirator. Decant the reaction mixture onto the filter paper, minimising any transfer of the solid aspirin.
9. Add 15 cm^3 of cold water to the flask, swirl, and cool again. Pour the solution mixture and the crystals of aspirin onto the filter paper. Repeat until the transfer of the crystals to the vacuum filter is complete. Wash the aspirin crystals on the filter paper with 10 cm^3 of ice water. Maintain the vacuum to dry the crystals as best as possible. Determine the mass of the crude aspirin crystals. [Alternative: simple filtration should be acceptable but essential details of filtration process need to be mentioned.]

Recrystallisation of Aspirin [Note: the process of recrystallisation has to be mentioned clearly with essential details.]

1. Place the aspirin crystals in a 100 cm³ beaker. Add 50 cm³ of water.
2. Warm the mixture in a 60°C water bath. Warm the mixture until all aspirin crystals dissolves.
3. Cover the beaker with a watch glass, remove it from the heat, and set it aside to cool slowly. Set the beaker in an ice/water bath. Crystals of aspirin will form.
4. Collect the aspirin by vacuum filtration. Wash the crystals with two 10 cm³ volumes of ice water. Maintain the vacuum to air dry the aspirin.
5. Transfer the dry aspirin crystals to a pre-weighed sample container or vial. Determine the mass of the aspirin crystals.

Determine the Melting Point of the Aspirin Sample to check for purity [Note: Heating aspirin to constant mass and measuring the yield as a way to test purity does not make sense. And cannot determine boiling point of aspirin, read question carefully!]

1. Fill a capillary melting point tube to a depth of 0.2 cm with the recrystallised aspirin.
2. Place the capillary tube in the melting point apparatus. Determine its melting point. If it melts sharply at 135 °C, the aspirin prepared is pure, otherwise the melting point will be observed to be lower, which implies impurities present.

(c) Identify **one** potential safety hazard in this experiment and state how you would minimise this risk. [1]

When ethanoic anhydride is in contact with skin, may develop rashes and burns since it is a corrosive liquid. Also 85% phosphoric acid is very concentrated. Avoid direct contact with such chemicals by wearing safety gloves.

OR

When mixing HOC₆H₄CO₂H and (CH₃CO)₂O, reactant mixture may spill over the round-bottomed flask since there will be initial violent reaction. Add (CH₃CO)₂O and 85% phosphoric acid dropwise over a minute to control the rate of reaction.

OR

Addition of 2 to 3 cm³ of water to hot reaction mixture to hydrolyse any unreacted ethanoic anhydride may cause mixture to boil and may cause splashing and spurting. Wear safety goggles throughout the experiment.

[Note: flammability of organic compounds is also another possible hazard. Any one of the above, note the hazards are given as clues in the question itself. Other answers not listed in question receive no credit.)

Example 2: Distinguishing tests to identify unknown (modified from PJC Prelim P2Q1)
(Suggested solutions in boxes)

Task: To determine the identity of four solutions.

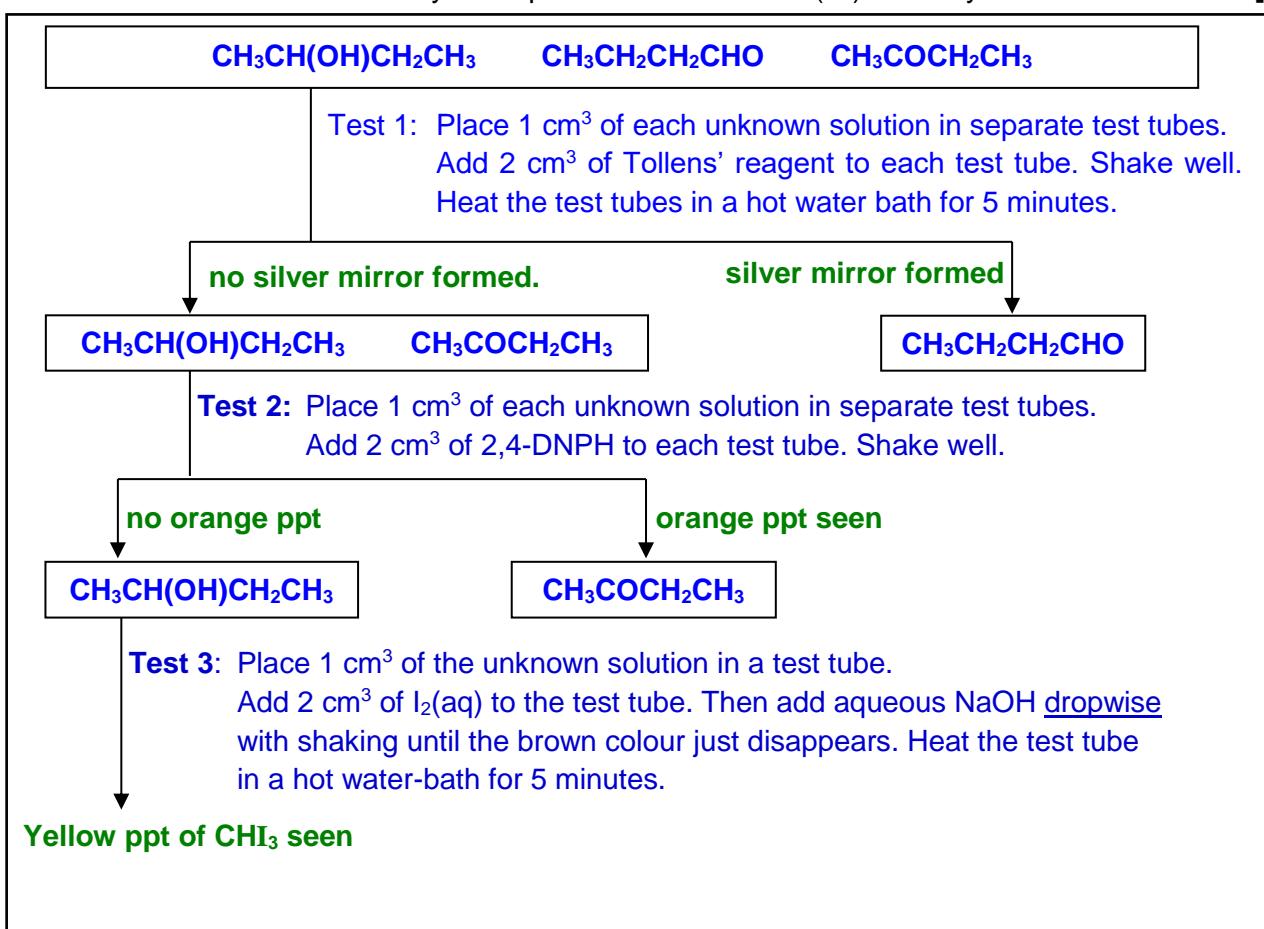
The labels of 4 bottles have fallen off. Each bottle contains **one** of the following:

- butan-2-ol
- butanal
- butanone
- an aqueous solution of iron(III) chloride

(a) State the reagents you would use, and observations you could make, to conclusively identify the iron(III) ions. You do **not** need to test for the other organic compounds. [2]

Reagents	Observations
aqueous phenol / $C_6H_5OH(aq)$	violet / purple complex / solution formed
aqueous sodium / potassium thiocyanate OR $NaSCN(aq)$ / $KSCN(aq)$	blood red solution / complex formed
$NaOH(aq)$ / $KOH(aq)$ / $NH_3(aq)$	reddish-brown ppt formed, insoluble in excess
[any one]	

(b) (i) Describe the procedures of simple chemical tests by which you could identify each of the organic substances. You are **not** allowed to identify the substances by elimination. You do **not** need to identify the aqueous solution of iron(III) ions anymore. [7]



- (ii) For the tests you proposed in (i), state and briefly explain any **one** issue of reliability or limitations that may be present. [1]

- **[If oxidation is performed]** Butan-2-ol needs to be identified before butanal since both can be oxidised, or vice-versa.
- **[if tri-iodomethane test is performed]** Butan-2-ol needs to be identified before butanone since both give yellow ppt, or vice-versa.
- **[if 2,4-DNPH is used]** Butanal needs to be identified before butanone since both give an orange ppt, or vice-versa.
- **[if sodium is used]** Ensure the unknowns are anhydrous, as water can react with sodium metal to give a false positive test.
[any one, with correct reagents used earlier]

- (c) State and explain **two** safety precautions employed in the course to distinguish the four unknowns given [2]

- Butan-2-ol, butanal and butanone are flammable.
Ensure no naked flame is used and a water bath is used for heating.
- Butan-2-ol, butanal and butanone are volatile and toxic.
Perform experiment in a fume cupboard to minimise fumes inhaled.
- Butan-2-ol, butanal and butanone is toxic (and corrosive).
Use test tube holder, add chemicals using droppers, wear gloves and goggles to avoid direct contact with chemicals.
- **[if sodium is used]** Hydrogen gas is explosive.
Ensures absence of naked flame / only used a small piece of sodium metal OR ensure anhydrous condition for sodium.

[any 2, reject precaution without reason]

[Total: 12]

7.2 Inorganic Qualitative Analysis

7.2.1 QUALITATIVE ANALYSIS NOTES (EXPLANATION)

It is important to take note of the significance of each reagent for each specific test. Each reagent used in a test has a specific role and you will have to identify that role in order to be able to record the expected observation and hence, the corresponding deduction. Through practice and familiarity, you will be able to do it.

Take some time to study the tests and understand what the various tests are meant for. Each specific test involving specific reagents is to be understood in terms of its function and what and why it is used for.

Chemical equations are also provided to give clarity to the reactions occurring. However, in the A Level Practical Test, no chemical equations are required, unless they are specifically asked for.

1. Reactions of aqueous cations

Cation	Reaction with	
	NaOH(aq)	NH ₃ (aq)
Aluminium, Al³⁺(aq)	White precipitate, soluble in excess. $\text{Al}^{3+}(\text{aq}) + 3\text{OH}^-(\text{aq}) \rightarrow \text{Al(OH)}_3(\text{s})$ $\text{Al(OH)}_3(\text{s}) + \text{OH}^-(\text{aq}) \rightarrow [\text{Al(OH)}_4]^- (\text{aq})$	White precipitate, insoluble in excess. $\text{Al}^{3+}(\text{aq}) + 3\text{OH}^-(\text{aq}) \rightarrow \text{Al(OH)}_3(\text{s})$
Zinc, Zn²⁺(aq)	White precipitate, soluble in excess. $\text{Zn}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Zn(OH)}_2(\text{s})$ $\text{Zn(OH)}_2(\text{s}) + 2\text{OH}^-(\text{aq}) \rightarrow [\text{Zn(OH)}_4]^{2-} (\text{aq})$	White precipitate, soluble in excess. $\text{Zn}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Zn(OH)}_2(\text{s})$ $\text{Zn(OH)}_2(\text{s}) + \text{NH}_3(\text{aq}) \rightarrow [\text{Zn(NH}_3)_4]^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq})$
Barium, Ba²⁺(aq)	No precipitate (if reagents are pure).	No precipitate.
Calcium, Ca²⁺(aq)	White precipitate (with high [Ca ²⁺]). $\text{Ca}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Ca(OH)}_2(\text{s})$	No precipitate.
Magnesium, Mg²⁺(aq)	White precipitate, insoluble in excess. $\text{Mg}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Mg(OH)}_2(\text{s})$	White precipitate, insoluble in excess. $\text{Mg}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Mg(OH)}_2(\text{s})$
Chromium(III), Cr³⁺(aq) (Green)	Grey-green precipitate, soluble in excess to give a dark green solution. $\text{Cr}^{3+}(\text{aq}) + 3\text{OH}^-(\text{aq}) \rightarrow \text{Cr(OH)}_3(\text{s})$ $\text{Cr(OH)}_3(\text{s}) + 3\text{OH}^-(\text{aq}) \rightarrow [\text{Cr(OH)}_6]^{3-}(\text{aq})$	Grey-green precipitate, insoluble in excess. $\text{Cr}^{3+}(\text{aq}) + 3\text{OH}^-(\text{aq}) \rightarrow \text{Cr(OH)}_3(\text{s})$

Cation	Reaction with	
	NaOH(aq)	NH ₃ (aq)
Copper(II), Cu ²⁺ (aq) (Blue)	Pale blue precipitate, insoluble in excess. $\text{Cu}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Cu}(\text{OH})_2(\text{s})$	Pale blue precipitate, soluble in excess to give a dark blue solution. $\text{Cu}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Cu}(\text{OH})_2(\text{s})$ $\text{Cu}(\text{OH})_2(\text{s}) + 4\text{NH}_3(\text{aq}) \rightarrow [\text{Cu}(\text{NH}_3)_4]^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq})$
Iron(II), Fe ²⁺ (aq) (Pale green)	Green precipitate, insoluble in excess. $\text{Fe}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Fe}(\text{OH})_2(\text{s})$ The green precipitate, Fe(OH) ₂ is rapidly oxidised to red-brown precipitate, Fe(OH) ₃ (s), on exposure to air. (Sometimes, the observation is a 'dirty green' precipitate is seen).	Green precipitate, insoluble in excess. $\text{Fe}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Fe}(\text{OH})_2(\text{s})$ The green precipitate, Fe(OH) ₂ is rapidly oxidised to red-brown precipitate, Fe(OH) ₃ (s), on exposure to air. (Sometimes, the observation is a 'dirty green' precipitate is seen).
Iron(III), Fe ³⁺ (aq) (Yellow)	Red-brown precipitate, insoluble in excess. $\text{Fe}^{3+}(\text{aq}) + 3\text{OH}^-(\text{aq}) \rightarrow \text{Fe}(\text{OH})_3(\text{s})$	Red-brown precipitate, insoluble in excess. $\text{Fe}^{3+}(\text{aq}) + 3\text{OH}^-(\text{aq}) \rightarrow \text{Fe}(\text{OH})_3(\text{s})$
Manganese(II), Mn ²⁺ (aq) (Pale pink)	Off-white precipitate, insoluble in excess. $\text{Mn}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Mn}(\text{OH})_2(\text{s})$ On exposure to air, the off-white precipitate is rapidly oxidised to the brown precipitate, Mn(OH) ₃ .	Off-white precipitate, insoluble in excess. $\text{Mn}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Mn}(\text{OH})_2(\text{s})$ On exposure to air, the off-white precipitate is rapidly oxidised to the brown precipitate, Mn(OH) ₃ .
Ammonium, NH ₄ ⁺ (aq)	No ppt. Ammonia gas, NH ₃ produced on heating. $\text{NH}_4^+(\text{aq}) + \text{OH}^-(\text{aq}) \rightarrow \text{NH}_3(\text{g}) + \text{H}_2\text{O}(\text{l})$	

Points of interest

- **NaOH** is a precipitating agent by supplying high concentration of OH⁻ as it is a strong base, fully ionised in solution ($\text{NaOH} \rightarrow \text{Na}^+ + \text{OH}^-$). So those insoluble hydroxides with high or low K_{sp} will be normally be precipitated.
- **NH₃(aq)** is also a precipitating agent by supplying lower concentration of OH⁻ as it is a weak base, only partially ionised in solution ($\text{NH}_3 + \text{H}_2\text{O} \rightleftharpoons \text{NH}_4^+ + \text{OH}^-$). So those insoluble hydroxides with low K_{sp} will be normally be precipitated. In addition, NH₃(aq) can also act as **ligands** in the formation of complex ions.
- Note that Zn²⁺ can be distinguished from Al³⁺ by using NH₃(aq) as Zn²⁺ gives a white precipitate with NH₃(aq), which is **soluble** in excess NH₃. For Al³⁺ it forms a white precipitate but the precipitate is **insoluble** in excess NH₃.
- Transition metal cations are easily identified as they give **coloured** precipitates with NaOH(aq) or NH₃(aq) or by their **coloured** aqueous solutions.
- All ammonium salts (NH₄⁺) react with NaOH(aq) to give **NH₃ gas**.

2. Reactions of anions

Devarda's alloy is a mixture of Al and Zn.

3. Test for gases

Gas	Test and test result
Carbon dioxide, CO ₂	Gives a white precipitate with limewater, Ca(OH) ₂ (aq). (Precipitate dissolves with excess CO ₂ gas). $\text{CO}_2(\text{g}) + \text{Ca}(\text{OH})_2(\text{aq}) \rightarrow \text{CaCO}_3(\text{s}) + \text{H}_2\text{O}(\text{g})$ $\text{CaCO}_3(\text{s}) + \text{CO}_2(\text{g}) + \text{H}_2\text{O}(\text{l}) \rightarrow \text{Ca}(\text{HCO}_3)_2(\text{aq})$
Ammonia, NH ₃	Turns damp red litmus paper blue (ammonia is a basic gas).
Oxygen, O ₂	Relights a glowing splint.
Hydrogen, H ₂	'Pops' with a lighted splint.
Chlorine, Cl ₂	Bleaches damp litmus paper.
Sulfur dioxide, SO ₂	Turns acidified K ₂ Cr ₂ O ₇ (aq) from orange to green or turns acidified KMnO₄(aq) from purple to colourless. $\text{Cr}_2\text{O}_7^{2-}(\text{aq}) + 3\text{SO}_2(\text{g}) + 2\text{H}^+(\text{aq}) \rightarrow 2\text{Cr}^{3+}(\text{aq}) + 3\text{SO}_4^{2-}(\text{aq}) + \text{H}_2\text{O}(\text{l})$ orange green $2\text{MnO}_4^-(\text{aq}) + 5\text{SO}_2(\text{g}) + 2\text{H}_2\text{O}(\text{l}) \rightarrow 2\text{Mn}^{2+}(\text{aq}) + 5\text{SO}_4^{2-}(\text{aq}) + 4\text{H}^+(\text{aq})$ purple colourless Note: In view of the fact that K ₂ Cr ₂ O ₇ is now discouraged in its use because of health reasons (it is cancer-causing(carcinogenic)), KMnO ₄ is the preferred choice.

4. Colour of halogens

Halogen	Colour of element	Colour in aqueous solution	Colour in hexane
Chlorine, Cl ₂	Greenish yellow gas	Pale yellow	Pale yellow
Bromine, Br ₂	Reddish brown gas/liquid	Orange	Orange-red
Iodine, I ₂	Black solid/purple gas	Brown	Purple

	TEST	OBSERVATION	DEDUCTION
(a)(i)	Add potassium iodide, KI(aq) to test sample, followed by addition of sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3(\text{aq})$.	Brown solution (sometimes, black solid at bottom of test tube), brown solution is decolourised.	Iodine liberated. Test sample is an oxidising agent (OA). I^- is oxidised to I_2 . I_2 is reduced to I^- by $\text{S}_2\text{O}_3^{2-}$.
		Oxidizing agent present, I_2 is reduced. $2\text{I}^-(\text{aq}) \rightarrow \text{I}_2 + 2\text{e}^-(\text{aq})$ $2\text{S}_2\text{O}_3^{2-}(\text{aq}) + \text{I}_2(\text{aq}) \rightarrow \text{S}_4\text{O}_6^{2-}(\text{aq}) + 2\text{I}^-(\text{aq})$ brown colourless	
(ii)	Add KI(aq) to test sample, followed by addition of $\text{Na}_2\text{S}_2\text{O}_3(\text{aq})$.	Cream/off white ppt in brown solution,	Iodine liberated. Test sample is an OA. I^- is oxidised to I_2 . $\text{Cu}^{2+}(\text{aq}) + 4\text{I}^-(\text{aq}) \rightarrow 2\text{CuI}(\text{s}) + \text{I}_2(\text{aq})$ cream ppt brown
		brown solution is decolourised leaving behind cream/off white ppt.	I_2 is reduced to I^- by $\text{S}_2\text{O}_3^{2-}$.
		$2\text{S}_2\text{O}_3^{2-}(\text{aq}) + \text{I}_2(\text{aq}) \rightarrow \text{S}_4\text{O}_6^{2-}(\text{aq}) + 2\text{I}^-(\text{aq})$ brown colourless	
(b)(i)	Add silver nitrate, AgNO₃(aq) to test sample followed by addition of dilute nitric acid, HNO_3 . Add AgNO ₃ (aq) to test sample followed by addition of NH ₃ (aq).	White ppt insoluble in acid. White ppt soluble in excess NH ₃ (aq) to give a colourless solution.	Cl^-
		$\text{Ag}^+(\text{aq}) + \text{Cl}^-(\text{aq}) \rightarrow \text{AgCl}(\text{s})$ white ppt $\text{AgCl}(\text{s}) + 2\text{NH}_3(\text{aq}) \rightarrow [\text{Ag}(\text{NH}_3)_2]^+\text{Cl}^-(\text{aq})$ Colourless	
(ii)	Add AgNO₃(aq) to test sample followed by addition of dilute HNO ₃ and then NH ₃ (aq).	Pale yellow/cream ppt insoluble in acid and sparingly soluble in NH ₃ (aq).	Br^-
		$\text{Ag}^+(\text{aq}) + \text{Br}^-(\text{aq}) \rightarrow \text{AgBr}(\text{s})$ cream ppt	
(iii)	Add AgNO₃(aq) to test sample followed by addition of dilute HNO ₃ and then NH ₃ (aq).	Yellow ppt insoluble in acid and insoluble in NH ₃ (aq).	I^-
		$\text{Ag}^+(\text{aq}) + \text{I}^-(\text{aq}) \rightarrow \text{AgI}(\text{s})$ yellow ppt	
(iv)	Add AgNO₃(aq) to test sample followed by addition of dilute HNO ₃ .	No ppt.	Absence of halides/ Cl^- , Br^- , I^- .

	TEST	OBSERVATION	DEDUCTION
(e)	Add barium chloride, $\text{BaCl}_2(\text{aq})/\text{Ba}(\text{NO}_3)_2$ to test sample followed by dilute hydrochloric acid, HCl/HNO_3 .	White ppt insoluble in acid. White ppt soluble in acid. No ppt.	SO_4^{2-} . SO_3^{2-} . Absence of SO_4^{2-} , SO_3^{2-} .
		$\text{Ba}^{2+}(\text{aq}) + \text{SO}_4^{2-} \rightarrow \text{BaSO}_4(\text{s})$ white ppt BaSO_4 is insoluble in acid.	
		$\text{Ba}^{2+}(\text{aq}) + \text{SO}_3^{2-} \rightarrow \text{BaSO}_3(\text{s})$ white ppt	
		BaSO_3 is soluble in acid. $\text{BaSO}_3(\text{s}) + 2\text{HCl}(\text{aq}) \rightarrow \text{BaCl}_2(\text{aq}) + \text{SO}_2(\text{g}) + \text{H}_2\text{O}(\text{l})$ soluble	
(f)	Add $\text{NaOH}(\text{aq})$ to test sample.	White ppt soluble in excess $\text{NaOH}(\text{aq})$.	Al^{3+} , Zn^{2+} .
		$\text{Al}^{3+}(\text{aq}) + 3\text{OH}^-(\text{aq}) \rightarrow \text{Al}(\text{OH})_3(\text{s})$ white ppt	
		$\text{Al}(\text{OH})_3(\text{s}) + \text{OH}^-(\text{aq}) \rightarrow [\text{Al}(\text{OH})_4]^- (\text{aq})$ soluble	
		$\text{Zn}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Zn}(\text{OH})_2(\text{s})$ white ppt	
		$\text{Zn}(\text{OH})_2(\text{s}) + 2\text{OH}^-(\text{aq}) \rightarrow [\text{Zn}(\text{OH})_4]^{2-} (\text{aq})$ soluble	
(g)	Add $\text{NaOH}(\text{aq})$ to test sample and heat gently.	No ppt. Upon heating, gas liberated turns damp red litmus paper blue.	NH_4^+ ? NH_3 gas liberated. NH_4^+ present.
		$\text{NH}_4^+(\text{s}) + \text{OH}^-(\text{aq}) \rightarrow \text{NH}_3(\text{g}) + \text{H}_2\text{O}(\text{l})$ All ammonium salts liberate NH_3 gas when heated with a base.	
		No ppt. Upon heating, no effect on damp red litmus paper.	NH_4^+ ? No NH_3 gas liberated. Absence of NH_4^+ .

	TEST	OBSERVATION	DEDUCTION
(h)	Add ammonia, NH₃(aq) to test sample.	White ppt soluble in excess NH ₃ (aq). $\text{NH}_3(\text{aq}) + \text{H}_2\text{O}(\text{l}) \rightleftharpoons \text{NH}_4^+(\text{aq}) + \text{OH}^-(\text{aq})$ $\text{Zn}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightleftharpoons \text{Zn}(\text{OH})_2(\text{s})$ white ppt $\text{Zn}(\text{OH})_2(\text{s}) + 4\text{NH}_3(\text{aq}) \rightarrow [\text{Zn}(\text{NH}_3)_4]^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq})$ soluble	Zn ²⁺ .
(i)	Add NH₃(aq) to test sample. Filter and add dilute H ₂ SO ₄ /HCl/HNO ₃ dropwise to the filtrate till in excess.	White ppt insoluble in excess NH ₃ (aq). Colourless filtrate. White ppt with acid, soluble in excess acid. $\text{NH}_3(\text{aq}) + \text{H}_2\text{O}(\text{l}) \rightleftharpoons \text{NH}_4^+(\text{aq}) + \text{OH}^-(\text{aq})$ Residue: $\text{Al}^{3+}(\text{aq}) + 3\text{OH}^-(\text{aq}) \rightarrow \text{Al}(\text{OH})_3(\text{s})$ white ppt Filtrate: $[\text{Zn}(\text{NH}_3)_4]^{2+}(\text{aq})$ $\text{Zn}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightleftharpoons \text{Zn}(\text{OH})_2(\text{s}) \rightleftharpoons [\text{Zn}(\text{NH}_3)_4]^{2+}(\text{aq})$ H ⁺ white ppt H ⁺ With H ₂ SO ₄ /HCl/HNO ₃ , Zn(OH) ₂ is formed, then with excess acid, soluble ZnSO ₄ /ZnCl ₂ /Zn(NO ₃) ₂ is formed as the positions of equilibrium above shift to the left.	A/ ³⁺ present in residue. Filtrate contains Zn ²⁺ . With H ₂ SO ₄ /HCl/HNO ₃ , Zn(OH) ₂ is formed, then with excess acid, soluble ZnSO ₄ /ZnCl ₂ /Zn(NO ₃) ₂ is formed as the positions of equilibrium above shift to the left.
(j)	Add NH₃(aq) to test sample. Then add aqueous or solid ammonium chloride, NH₄Cl .	White ppt. White ppt dissolves in the presence of NH ₄ Cl. $\text{NH}_3(\text{aq}) + \text{H}_2\text{O}(\text{l}) \rightleftharpoons \text{NH}_4^+(\text{aq}) + \text{OH}^-(\text{aq})$ $\text{NH}_4\text{Cl}(\text{aq}) \rightarrow \text{NH}_4^+(\text{aq}) + \text{Cl}^-(\text{aq})$ NH ₄ Cl provides a large excess of NH ₄ ⁺ , therefore position of equilibrium above shifts to left due to the common ion effect. So, [OH⁻] decreases . So, hydroxides of high K_{sp} will not be precipitated as [OH ⁻] is too low to exceed the K _{sp} of the hydroxides.	Hydroxides of high K_{sp} suspected, e.g., Mg ²⁺ .
(k)	Add dilute H ₂ SO ₄ to test sample followed by iron(II) ammonium sulfate or freshly prepared iron(II) sulfate.	Green solution turns yellow or brownish-yellow. $\text{Fe}^{2+}(\text{aq}) \rightarrow \text{Fe}^{3+}(\text{aq})$ green yellow No change in colour.	Test sample is an oxidising agent (OA). Fe ²⁺ is oxidised to Fe ³⁺ . OA $\text{Fe}^{2+}(\text{aq}) \rightarrow \text{Fe}^{3+}(\text{aq})$ green yellow Test sample is not an OA.
(l)	Add dilute H ₂ SO ₄ to test sample followed by potassium manganate(VII), KMnO₄(aq) .	Purple MnO ₄ ⁻ /H ⁺ is decolourised. $\text{MnO}_4^-(\text{aq}) + 8\text{H}^+(\text{aq}) + 5\text{e}^- \rightarrow \text{Mn}^{2+}(\text{aq}) + 4\text{H}_2\text{O}(\text{l})$ purple colourless Purple MnO ₄ ⁻ /H ⁺ is not decolourised.	Test sample is a reducing agent (RA). MnO ₄ ⁻ (aq) + 8H ⁺ (aq) + 5e ⁻ → Mn ²⁺ (aq) + 4H ₂ O(l) purple colourless Test sample is not a RA.

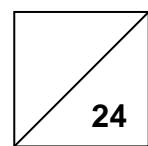
	TEST	OBSERVATION	DEDUCTION
(m)	Add sodium sulphite, $\text{Na}_2\text{SO}_3(\text{aq})$ to test sample followed by $\text{BaCl}_2(\text{aq})$. Add dilute HCl to the resulting mixture.	White ppt insoluble in acid.	SO_4^{2-} . SO_3^{2-} is oxidised to SO_4^{2-} . Test sample is an OA.
		OA $\text{SO}_3^{2-}(\text{aq}) \rightarrow \text{SO}_4^{2-}(\text{aq})$ $\text{Ba}^{2+}(\text{aq}) + \text{SO}_4^{2-}(\text{aq}) \rightarrow \text{BaSO}_4(\text{s})$ white ppt, insoluble in acid	
(n)	Add conc HCl to test sample and heat.	Gas liberated bleaches damp litmus paper.	Cl_2 liberated; test sample is an OA. HCl is oxidised to Cl_2 .
		N.B. Do not report and test for HCl! OA $\text{HCl} \rightarrow \text{Cl}_2$	
(o)	Add concentrated H_2SO_4 to test sample and heat.	Brown fumes liberated.	Br_2 or NO_2 liberated. Br^- or NO_3^- present.
		$\text{H}_2\text{SO}_4(\text{l}) + \text{Br}^-(\text{s}) \rightarrow \text{HSO}_4^-(\text{aq}) + \text{HBr}(\text{g})$ $2\text{HBr}(\text{g}) + \text{H}_2\text{SO}_4(\text{l}) \rightarrow \text{SO}_2(\text{g}) + 2\text{H}_2\text{O}(\text{l}) + \text{Br}_2(\text{g})$ $\text{H}_2\text{SO}_4(\text{l}) + \text{NO}_3^-(\text{aq}) \rightarrow \text{HSO}_4^-(\text{aq}) + \text{HNO}_3(\text{l})$ $2\text{HNO}_3(\text{l}) \rightarrow 2\text{NO}_2(\text{g}) + \frac{1}{2}\text{O}_2(\text{g}) + \text{H}_2\text{O}(\text{l})$	
		Gas which has a vinegar smell liberated.	$\text{CH}_3\text{CO}_2\text{H}$ vapour liberated. CH_3CO_2^- present.
		$\text{H}_2\text{SO}_4(\text{l}) + \text{CH}_3\text{CO}_2^-(\text{s}) \rightarrow \text{HSO}_4^-(\text{aq}) + \text{CH}_3\text{CO}_2\text{H}(\text{g})$	
(p)	Add solid sodium carbonate, Na_2CO_3 /sodium hydrogen carbonate, NaHCO_3 to test sample.	Effervescence. Gas liberated gives a white ppt with limewater.	CO_2 liberated. Test sample is an acid/acidic.
		$2\text{H}^+(\text{aq}) + \text{CO}_3^{2-}(\text{s}) \rightarrow \text{CO}_2(\text{g}) + \text{H}_2\text{O}(\text{l})$ $\text{H}^+(\text{aq}) + \text{HCO}_3^-(\text{s}) \rightarrow \text{CO}_2(\text{g}) + \text{H}_2\text{O}(\text{l})$	
(q)	Add magnesium, Mg ribbon to test sample.	Effervescence. Gas liberated gives a 'pop' sound with a lighted splinter.	H_2 liberated. Test sample is an acid/acidic.
		$2\text{H}^+(\text{aq}) + \text{Mg}(\text{s}) \rightarrow \text{Mg}^{2+}(\text{aq}) + \text{H}_2(\text{g})$	
(r)	Add solid Na_2SO_3 to test sample and heat cautiously.	Gas liberated decolourises purple acidified $\text{KMnO}_4(\text{aq})$.	SO_2 liberated. Test sample is an acid/acidic.
		$2\text{H}^+(\text{aq}) + \text{SO}_3^{2-}(\text{s}) \rightarrow \text{SO}_2(\text{g}) + \text{H}_2\text{O}(\text{l})$	

	TEST	OBSERVATION	DEDUCTION
(s)	Add copper(II) sulfate, $\text{CuSO}_4(\text{aq})$ to test sample.	Blue colour turns green. $[\text{Cu}(\text{H}_2\text{O})_6]^{2+}(\text{aq}) + 4\text{Cl}^- (\text{aq}) \rightleftharpoons \text{CuCl}_4^{2-} (\text{aq}) + 6\text{H}_2\text{O}(\text{l})$ blue yellow blue + yellow = green!	Concentrated HCl or Cl^- .
(t)	Add $\text{CuSO}_4(\text{aq})$ to test sample followed by $\text{Na}_2\text{S}_2\text{O}_3(\text{aq})$.	Cream ppt/off white ppt in a brown solution. With $\text{S}_2\text{O}_3^{2-}$, brown solution is decolourised leaving behind the cream/off white ppt.	I_2 liberated. I^- present in test sample. I^- is oxidised by Cu^{2+} to I_2 . $\text{S}_2\text{O}_3^{2-}$ reduces I_2 to I^- .

Name: (2T.....)

Date:

**Catholic Junior College
H2 Chemistry 9729 (JC2)**



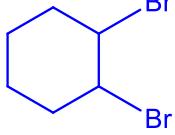
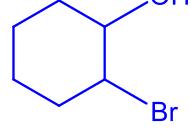
Experiment 15: Organic Qualitative Analysis 1

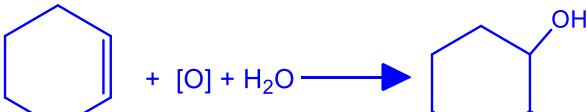
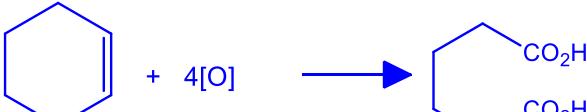
Part 1: Typical reactions of alkenes and halogenoalkanes.

The characteristic properties of organic compounds are determined by their functional groups. In this activity, you will explore some typical reactions associated with the alkene functional group.

Caution: Do **not** heat any test-tube with a naked flame. Use a water-bath (250 cm³ beaker half-filled with hot water) instead. It is essential that you **replace the lid as soon as you have removed the organic chemical** for each test.

Alkenes

Test	Observations	Questions
<p>1) Add 1 cm depth of cyclohexene to a test-tube. Add 1-2 drops of bromine water /aqueous bromine. to this test-tube.</p> <p><i>Do not add excess aqueous bromine.</i> (Take care as bromine water is corrosive.)</p> 	<p>Orange Br₂(aq) is decolourised (turn colourless). [1]</p> 	<p>(a) Name the type of reaction that has occurred. electrophilic addition[1]</p> <p>(b) Name the two possible organic products and draw the structures.</p> <p> </p> <p>1,2-dibromocyclohexane 2-bromocyclohexanol</p> <p>[1 both structures] [1 both correct names]</p>
	<p>Electrophilic Addition of Alkene with Bromine Water https://www.youtube.com/watch?v=1yXQ8QA_8bk</p> <p>In the video: Orange Br₂ decolorises when added to the alkene, but remains orange when added to the alkane.</p> 	

Test	Observations	Questions
<p>2) Add 1 cm depth of cyclohexene to a test-tube.</p> <p>Add 1 cm depth of aqueous sodium hydroxide, followed by 1-2 drops of potassium manganate(VII)(aq) to the test-tube.</p> <p><i>Do not add excess potassium manganate(VII).</i></p>	<p>A green solution is seen and on standing a brown ppt is obtained. [1] (Brown ppt is MnO₂)</p>	<p>(a) Name the type of reaction that has occurred. Oxidative addition. [1] (Mild oxidation)</p> <p>(b) Write a balanced equation for this reaction, using [O] to represent KMnO₄.</p> <p style="text-align: center;">  [1] </p> <p>Oxidation using KMnO₄/NaOH https://www.youtube.com/watch?v=dIXEkSE7eG8</p> <p>In the video: When Na₂SO₃ (sodium sulfite) is added to an alkaline solution of KMnO₄, it gets oxidised. The purple KMnO₄ turns green at first (due to formation of MnO₄²⁻), then slowly decolorises as a brown ppt of MnO₂ forms.</p> <p style="text-align: right;"></p>
<p>3) Add 1 cm depth of cyclohexene to a test-tube.</p> <p>Add 1 cm depth of aqueous sulfuric acid, followed by 1-2 drops of potassium manganate(VII) (aq) to this test-tube.</p> <p>Leave the test tube to stand in a hot water bath.</p> <p><i>Do not add excess potassium manganate(VII).</i></p>	<p>Purple KMnO₄ is decolourised (Colourless solution) on heating. [1]</p>	<p>(a) Name the type of reaction that has occurred. Oxidative cleavage. [1] (Strong oxidation)</p> <p>(b) Write a balanced equation for this reaction, using [O] to represent KMnO₄.</p> <p style="text-align: center;">  [1] </p>

[10]

Halogenoalkanes

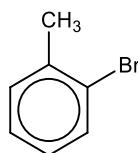
Solutions **A**, **B** and **C** are different halogenoalkanes (chloroethane, bromoethane and iodoethane, but not necessarily in that order).

Procedure	Observations	Questions
<p>1) Add 1cm depth of solution A to a test-tube.</p> <p>Add 1 cm depth of aqueous sodium hydroxide to this test-tube.</p> <p>Leave the test-tube to stand in a hot water bath for about 5 minutes.</p> <p>Remove the test-tube from the water bath and place it in the test-tube rack to cool to room temperature.</p> <p>Add 2 cm depth of aqueous nitric acid to the test-tube.</p> <p>Add 2-3 drops of aqueous silver nitrate to the test-tube.</p>	<p>Cream ppt [1]of AgBr</p> 	<p>a) State the identities of A, B and C.</p> <p>A : Bromoethane</p> <p>B : Iodoethane</p> <p>C : Chloroethane [1 for all 3, e.c.f]</p> <p>b) State the type of reaction with aqueous sodium hydroxide, and give the displayed structure of the organic product.</p> <p>Nucleophilic substitution [1] (S_N2 in this case, as all are primary halogenoalkanes)</p> <p style="text-align: center;"> $\begin{array}{c} \text{H} & & & \text{H} \\ & \diagdown & & \diagup \\ & \text{O} & & \\ & \diagup & & \diagdown \\ \text{H}-\text{C} & -\text{C}-\text{H} \\ & & \\ \text{H} & & \text{H} \end{array}$ [1] </p> <p>c) State the type of reaction with aqueous nitric acid, and suggest why the addition of nitric acid is necessary.</p>
2) Repeat the test in 1) with solution B .	yellow ppt [1]of AgI 	<p>Acid-base reaction. [1]</p> <p>To <u>remove any unreacted NaOH(aq) [1]</u>. If this is not done, NaOH would react with AgNO₃ in step (iii) to give a brown ppt of Ag₂O[1], which would mask the colour of the actual silver halide precipitate (AgX).</p>
3) Repeat the test in 1) with solution C .	white ppt [1]of AgCl 	
<p>4) Add 1 cm depth of aqueous sodium hydroxide to a test-tube.</p> <p>Add 2-3 drops of aqueous silver nitrate to the test-tube.</p>	Brown ppt [1]of Ag₂O	

Part 2: Planning

Consider the following three organic compounds.

hex-2-ene



2-bromopropane

Plan an investigation, using test-tube reactions, which would allow you to identify each of these three organic compounds.

Each compound should be identified by at least one positive test result. It is not sufficient to identify a compound simply by eliminating all the others.

Your plan should include:

- details of the reagents and conditions to be used,
- an outline of the sequence of steps you would follow,
- an explanation of how you would analyse your results in order to identify each compound.

Once a compound has been clearly identified, your plan should concentrate on distinguishing the remaining compounds.

Tests should be bromine (aq); warming with acidified potassium manganate (VII); use of NaOH, HNO₃ followed by AgNO₃

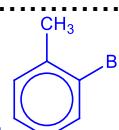
1. Add 1 cm depth of the 3 compounds **separately** to 3 test-tubes/boiling tubes.
2. Then add 1 cm depth of NaOH(aq) to each of these test-tubes/boiling tubes. Leave the test-tubes to stand in a **hot water bath** for about 5 minutes. Allow to cool, then add 2 cm depth of HNO₃(aq) to each test-tube, followed by the addition of 2 to 3 drops of AgNO₃(aq).

A cream ppt. seen in the test-tube that had 2-bromopropane. [1]

3. Repeat step 1 for the **remaining 2 unknown compounds** then add 1-2 drops of Br₂(aq) to these test-tubes.

Orange bromine is decolourised in the test-tube that had hexene. [1]

4. Repeat step 1 for the last compound. Add 1 cm depth of H₂SO₄ (aq), followed by 1-2 drops of KMnO₄ (aq) to this test-tube. Leave the test tube to stand in a **hot water bath**.



Purple KMnO₄ is decolourised, confirming that [1] is present. [1]

Mention of test-tube/boiling tube and Hot water bath. [1]

[4]

Name: (2T) Date:

**Catholic Junior College
H2 Chemistry (9729)**

Experiment 16: Inorganic Qualitative Analysis 1

Answer **ALL** the questions in the SPACES provided.

Part 1: Reactions of cations with NaOH(aq) and NH₃(aq)

Part 2: Reactions of anions with AgNO₃(aq), Ba(NO₃)₂(aq) and HCl(aq)

Part 3: Reactions to identify given cations and anions

In all the experiments, you are to follow the procedure as instructed, record the results of your experiments and then answer the questions that follow, where applicable.

You are provided with the **Qualitative Analysis notes** for reference. Use them to check with the observations which you have made.

Part 1

QUALITATIVE ANALYSIS

Reactions of cations with NaOH(aq) and NH₃(aq)

Procedure:

To a small portion (about 1 cm³) of each of the following solutions containing the **cation**, in a test-tube, add aqueous **sodium hydroxide** slowly (dropwise initially) until ***excess** has been added and no further change is observed.

Observation:

Record your **observations**, especially if a precipitate is formed, its colour and whether it is soluble or insoluble in **excess** of the reagent and any gas evolved, if applicable.

* How much is considered **excess**? It depends on how much precipitate is formed. **Sometimes**, it helps to **pour some of the precipitate** away before adding more reagent.

Repeat the procedure with aqueous **ammonia**.

For all positive observations, write **balanced chemical equations** for the reactions occurring.

Aqueous solution of	Results with NaOH(aq)	Results with NH ₃ (aq)
1 Magnesium sulfate $Mg^{2+}(aq)$	White precipitate, insoluble in excess. $Mg^{2+}(aq) + 2OH^-(aq) \rightarrow Mg(OH)_2(s)$	White precipitate, insoluble in excess. $Mg^{2+}(aq) + 2OH^-(aq) \rightarrow Mg(OH)_2(s)$
2 Calcium chloride $Ca^{2+}(aq)$	White precipitate (with high [Ca²⁺]). $Ca^{2+}(aq) + 2OH^-(aq) \rightarrow Ca(OH)_2(s)$	No precipitate.

Aqueous solution of	Results with NaOH(aq)	Results with NH ₃ (aq)
3 Barium chloride Ba ²⁺ (aq)	No precipitate (if reagents are pure).	No precipitate.
4 Aluminium sulfate Al ³⁺ (aq)	White precipitate, soluble in excess. $\text{Al}^{3+}(\text{aq}) + 3\text{OH}^-(\text{aq}) \rightarrow \text{Al}(\text{OH})_3(\text{s})$ $\text{Al}(\text{OH})_3(\text{s}) + \text{OH}^-(\text{aq}) \rightarrow [\text{Al}(\text{OH})_4]^- (\text{aq})$	White precipitate, insoluble in excess. $\text{Al}^{3+}(\text{aq}) + 3\text{OH}^-(\text{aq}) \rightarrow \text{Al}(\text{OH})_3(\text{s})$
5 Zinc sulfate Zn ²⁺ (aq)	White precipitate, soluble in excess. $\text{Zn}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Zn}(\text{OH})_2(\text{s})$ $\text{Zn}(\text{OH})_2(\text{s}) + 2\text{OH}^-(\text{aq}) \rightarrow [\text{Zn}(\text{OH})_4]^{2-}(\text{aq})$	White precipitate, soluble in excess. $\text{Zn}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Zn}(\text{OH})_2(\text{s})$ $\text{Zn}(\text{OH})_2(\text{s}) + \text{NH}_3(\text{aq}) \rightarrow [\text{Zn}(\text{NH}_3)_4]^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq})$
6 Manganese(II) sulfate Mn ²⁺ (aq)	Off-white precipitate, insoluble in excess. $\text{Mn}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Mn}(\text{OH})_2(\text{s})$ On exposure to air, the off-white precipitate is rapidly oxidised to the brown precipitate, Mn(OH) ₃ .	Off-white precipitate, insoluble in excess. $\text{Mn}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Mn}(\text{OH})_2(\text{s})$ On exposure to air, the off-white precipitate is rapidly oxidised to the brown precipitate, Mn(OH) ₃ .
7 Iron(II) sulfate Fe ²⁺ (aq)	Green precipitate, insoluble in excess. $\text{Fe}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Fe}(\text{OH})_2(\text{s})$ The green precipitate, Fe(OH) ₂ is rapidly oxidised to red-brown precipitate, Fe(OH) ₃ (s), on exposure to air. (Sometimes, the observation is a 'dirty green' precipitate is seen).	Green precipitate, insoluble in excess. $\text{Fe}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Fe}(\text{OH})_2(\text{s})$ The green precipitate, Fe(OH) ₂ is rapidly oxidised to red-brown precipitate, Fe(OH) ₃ (s), on exposure to air. (Sometimes, the observation is a 'dirty green' precipitate is seen).
8 Iron(III) chloride Fe ³⁺ (aq)	Red-brown precipitate, insoluble in excess. $\text{Fe}^{3+}(\text{aq}) + 3\text{OH}^-(\text{aq}) \rightarrow \text{Fe}(\text{OH})_3(\text{s})$	Red-brown precipitate, insoluble in excess. $\text{Fe}^{3+}(\text{aq}) + 3\text{OH}^-(\text{aq}) \rightarrow \text{Fe}(\text{OH})_3(\text{s})$

Aqueous solution of	Results with NaOH(aq)	Results with NH ₃ (aq)
9 Copper(II) sulfate Cu ²⁺ (aq)	Pale blue precipitate, insoluble in excess. $\text{Cu}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Cu}(\text{OH})_2(\text{s})$	Pale blue precipitate, soluble in excess to give a dark blue solution. $\text{Cu}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Cu}(\text{OH})_2(\text{s})$ $\text{Cu}(\text{OH})_2(\text{s}) + 4\text{NH}_3(\text{aq}) \rightarrow [\text{Cu}(\text{NH}_3)_4]^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq})$
10 Chromium(III) sulfate Cr ³⁺ (aq)	Grey-green precipitate, soluble in excess to give a dark green solution. $\text{Cr}^{3+}(\text{aq}) + 3\text{OH}^-(\text{aq}) \rightarrow \text{Cr}(\text{OH})_3(\text{s})$ $\text{Cr}(\text{OH})_3(\text{s}) + 3\text{OH}^-(\text{aq}) \rightarrow [\text{Cr}(\text{OH})_6]^{3-}(\text{aq})$	Grey-green precipitate, insoluble in excess. $\text{Cr}^{3+}(\text{aq}) + 3\text{OH}^-(\text{aq}) \rightarrow \text{Cr}(\text{OH})_3(\text{s})$
11 Ammonium chloride NH ₄ ⁺ (aq)	Heat gently after addition of NaOH(aq). No ppt. Ammonia gas, NH₃ produced on heating as it turns damp red litmus blue. $\text{NH}_4^+(\text{aq}) + \text{OH}^-(\text{aq}) \rightarrow \text{NH}_3(\text{g}) + \text{H}_2\text{O}(\text{l})$	

Part 2

QUALITATIVE ANALYSIS

Reactions of anions with $\text{AgNO}_3\text{(aq)}$, $\text{Ba}(\text{NO}_3)_2\text{(aq)}$ and $\text{HCl}\text{(aq)}$

Procedure:

To a small portion (about 1 cm³) of each of the following solutions containing the **anion**, in a test-tube, add aqueous **silver nitrate**. Mix well.

Observation:

Record your **observations**, especially if a precipitate is formed, its colour or any gas evolved, if applicable.

Repeat the procedure with aqueous **barium nitrate** and dilute aqueous **HCl**, where relevant. For all positive observations, write balanced chemical equations for the reactions occurring.

Aqueous solution of	Results with $\text{Ag}^+\text{(aq)}$	
1 Potassium chloride $\text{Cl}^-\text{(aq)}$	<p>Gives white precipitate with $\text{Ag}^+\text{(aq)}$, soluble in excess $\text{NH}_3\text{(aq)}$.</p> $\text{Ag}^+\text{(aq)} + \text{Cl}^-\text{(aq)} \rightarrow \text{AgCl(s)}$ $\text{AgCl(s)} + 2\text{NH}_3\text{(aq)} \rightarrow [\text{Ag}(\text{NH}_3)_2]^+\text{(aq)}$	
2 Potassium bromide $\text{Br}^-\text{(aq)}$	<p>Gives cream precipitate with $\text{Ag}^+\text{(aq)}$, partially soluble in excess $\text{NH}_3\text{(aq)}$.</p> $\text{Ag}^+\text{(aq)} + \text{Br}^-\text{(aq)} \rightarrow \text{AgBr(s)}$	
3 Potassium iodide $\text{I}^-\text{(aq)}$	<p>Gives yellow precipitate with $\text{Ag}^+\text{(aq)}$, insoluble in excess $\text{NH}_3\text{(aq)}$.</p> $\text{Ag}^+\text{(aq)} + \text{I}^-\text{(aq)} \rightarrow \text{AgI(s)}$	
4 Sodium sulfate(VI) $\text{SO}_4^{2-}\text{(aq)}$		
Aqueous solution of	Results with $\text{Ba}^{2+}\text{(aq)}$	Results with $\text{HCl}\text{(aq)}$ or dilute strong acids
1 Sodium sulfate(IV) or sodium sulfite $\text{SO}_3^{2-}\text{(aq)}$	<p>Add dilute $\text{HCl}\text{(aq)}$ to the resulting mixture, after addition of $\text{Ba}^{2+}\text{(aq)}$.</p> <p>White precipitate, soluble in dilute $\text{HCl}\text{(aq)}$, liberating SO_2 as it turns acidified $\text{KMnO}_4\text{(aq)}$ from purple to colourless.</p> $\text{Ba}^{2+}\text{(aq)} + \text{SO}_3^{2-}\text{(aq)} \rightarrow \text{BaSO}_3\text{(s)}$ $\text{SO}_3^{2-}\text{(s)} + 2\text{H}^+\text{(aq)} \rightarrow \text{SO}_2\text{(g)} + \text{H}_2\text{O(l)}$	

2 Sodium sulfate(VI) SO_4^{2-} (aq)	Add dilute HCl (aq) to the resulting mixture, after addition of Ba^{2+} (aq). White precipitate, insoluble in dilute HCl(aq). $\text{Ba}^{2+}\text{(aq)} + \text{SO}_4^{2-}\text{(aq)} \rightarrow \text{BaSO}_4\text{(s)}$	
3 Sodium carbonate CO_3^{2-} (aq)		Effervescence of CO_2 liberated by dilute HCl(aq) as it gives a white ppt with limewater. $\text{CO}_3^{2-} + \text{H}^+\text{(aq)} \rightarrow \text{CO}_2\text{(g)} + \text{H}_2\text{O(l)}$
Aqueous solution of	Add NaOH(aq) followed by Al foil or Devardar's alloy and heat. Record the observation	
1 Sodium nitrate(V) NO_3^- (aq)	NH_3 gas liberated on heating with NaOH and Al foil (or Devarda's alloy) as it turns damp red litmus blue. $3\text{NO}_3^-\text{(aq)} + 8\text{Al(s)} + 5\text{OH}^-\text{(aq)} + 18\text{H}_2\text{O(l)} \rightarrow 3\text{NH}_3\text{(g)} + 8[\text{Al(OH)}_4]^-$ (Al in a basic medium is a reducing agent, reduces NO_3^- to NH_3 gas).	
2 Sodium nitrate(III) or sodium nitrite NO_2^- (aq)	NH_3 gas liberated on heating with NaOH and Al foil (or Devarda's alloy) as it turns damp red litmus blue. $\text{NO}_2^-\text{(aq)} + 2\text{Al(s)} + \text{OH}^-\text{(aq)} + 5\text{H}_2\text{O(l)} \rightarrow \text{NH}_3\text{(g)} + 2[\text{Al(OH)}_4]^-$ (Al in a basic medium is a reducing agent, reduces NO_2^- to NH_3 gas). Note: Nitrogen(II) oxide, NO gas liberated with dilute strong acids, HCl(aq). $\text{NO}_2^-\text{(aq)} + \text{H}^+\text{(aq)} \rightarrow \text{NO(g)} + \text{H}_2\text{O(l)}$ (colourless NO gives pale brown NO_2 gas in air).	

Part 3

QUALITATIVE ANALYSIS

Reactions to identify given cations and anions

Procedure:

To a small portion (about 1 cm³) of the solution containing the cation or anion, in a test-tube, add the reagent gradually (dropwise initially) until no further change is observed.

Observation:

- Note the colour of precipitate and solubility of precipitate in ***excess** reagent.
- Note the evolution of NH₃ and the test used to identify the gas, if applicable.

Note that **NH₃ gas** is liberated when an ammonium (NH₄⁺) salt is heated with aqueous sodium hydroxide. NH₄⁺(s) + OH⁻(aq) → NH₃(g) + H₂O(l)

* How much is considered **excess**? It depends on how much precipitate is formed. Sometimes, it helps to pour some of the precipitate away before adding more reagent.

- (a) You are provided with **five colourless** solutions labelled **FC1, FC2, FC3, FC4, and FC5**. Each of the solutions contains one of the cations, NH₄⁺, Ba²⁺, Ca²⁺, Mg²⁺ and Zn²⁺ but **not necessarily** in this order.

Carry out the following tests to identify the **cation** present in each of the solutions **FC1, FC2, FC3, FC4, and FC5**. Record your observations in the table below.

	Add aqueous sodium hydroxide slowly until excess. Heat if necessary.	Add aqueous ammonia slowly until excess.	Identity of cation
FC1	No ppt. NH ₃ gas liberated when heated as it turns damp red litmus blue.	No ppt.	NH ₄ ⁺
FC2	White ppt insoluble in excess.	No ppt.	Ca ²⁺
FC3	No ppt.	No ppt.	Ba ²⁺
FC4	White ppt insoluble in excess.	White ppt insoluble in excess.	Mg ²⁺
FC5	White ppt soluble in excess	White ppt soluble in excess.	Zn ²⁺

(b) You are provided with **four coloured** solutions labelled **FC6, FC7, FC8** and **FC9**.

Each of the solutions contains one of the cations, Cr^{3+} , Cu^{2+} , Fe^{2+} and Mn^{2+} but **not necessarily** in this order.

Carry out the following tests to identify the **cation** present in each of the solutions **FC6, FC7, FC8** and **FC9**.

Record your observations in the table below. Do **not** discard the reaction mixtures.

	Colour of aqueous solution	Add aqueous sodium hydroxide slowly until excess.	Identity of cation
FC6	Blue	Pale blue ppt insoluble in excess.	Cu^{2+}
FC7	Pale green	Dirty green ppt insoluble in excess.	Fe^{2+}
FC8	Green/bluish green	Grey-green ppt soluble in excess to give a dark green solution.	Cr^{3+}
FC9	Pale pink (almost colourless)	White ppt/off-white ppt turning brown rapidly, insoluble in excess.	Mn^{2+}

What other observations can distinguish between solutions containing Cr^{3+} and Fe^{2+} ? Explain your answer.

Dirty green ppt of $\text{Fe}^{2+}(\text{aq})$ turns reddish-brown on standing in air as the dirty green ppt, Fe(OH)_2 is oxidised by O_2 (present in air) to Fe(OH)_3 , a reddish-brown ppt.

Dark green solution of Cr^{3+} remains unchanged as it is not oxidised.

- (c) You are provided with three solutions labelled **FA1**, **FA2** and **FA3**. Each of the solutions contains one of the anions, Cl^- , Br^- and I^- but **not** necessarily in this order.

By referring to the ***Qualitative Analysis Notes***, suggest a reagent or reagents needed to test for the presence of Cl^- , Br^- and I^- ions.

Add $\text{AgNO}_3(\text{aq})$ to FA1, FA2 and FA3 separately.

Carry out the test you have suggested to identify the anion present in each of the solutions, **FA1**, **FA2** and **FA3**. Record your observations in the table below.

	Observation	Identity of Anion
FA1	White ppt.	Cl^-
FA2	Yellow ppt.	I^-
FA3	Pale yellow / cream ppt.	Br^-

- (d) You are provided with two solutions labelled **FA4** and **FA5**. Each of the solutions contains one of the anions NO_2^- and NO_3^- but **not** necessarily in this order.

By referring to the ***Qualitative Analysis Notes***, suggest a reagent or reagents needed to test for the presence of NO_2^- and NO_3^- ions.

Add dilute hydrochloric acid to FA4, and FA5 separately.

Carry out the test you have suggested to identify the anion present in each of the solutions, **FA4** and **FA5**. Record your observations in the table below.

	Observation	Identity of Anion
FA4	No brown gas observed.	NO_3^-
FA5	Brown gas evolved.	NO_2^-

- (e) You are provided with two solutions labelled **FA6** and **FA7**. Each of the solutions contains one of the anions SO_4^{2-} and SO_3^{2-} but **not** necessarily in this order.

By referring to the ***Qualitative Analysis Notes***, suggest a reagent or reagents needed to test for the presence of SO_4^{2-} and SO_3^{2-} ions.

Add $\text{BaCl}_2(\text{aq})$, followed by dilute hydrochloric acid, separately to FA6 and FA7.

Carry out the test you have suggested to identify the anion present in each of the solutions, **FA6** and **FA7**. Record your observations in the table below.

	Observation	Identity of Anion
FA6	White ppt soluble in dilute hydrochloric acid to give a colourless solution.	SO_3^{2-}
FA7	White ppt insoluble in dilute hydrochloric acid.	SO_4^{2-}

- (f) You are provided with two solutions labelled **FA8** and **FA9**. Each of the solutions contains one of the anions CO_3^{2-} and CrO_4^{2-} but **not** necessarily in this order.

By referring to the ***Qualitative Analysis Notes***, suggest a reagent or reagents needed to test for the presence of CO_3^{2-} and CrO_4^{2-} ions.

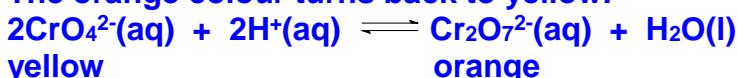
Add dilute hydrochloric acid to FA8 and FA9 separately.

Carry out the test you have suggested to identify the anion present in each of the solutions, **FA8** and **FA9**. Record your observations in the table below.

	Observation	Identity of Anion
FA8	Effervescence of CO_2 gas evolved as it gives a white ppt with limewater.	CO_3^{2-}
FA9	You are not required to carry out this test. The observation is given to you as follows. Yellow colour turns orange.	CrO_4^{2-}

What can be observed if $\text{NaOH}(\text{aq})$ is now added to the resulting solution of **FA9** above? You are not required to carry out this test. Explain clearly your answer with an aid of equation.

The orange colour turns back to yellow.



Addition of the base, $\text{NaOH}(\text{aq})$ removes H^+ in the solution so the POE shifts left. The yellow $\text{CrO}_4^{2-}(\text{aq})$ predominates.

Name: (2T)

Date:

Practical Worksheet 17: Organic Analysis 2

Pre-Lab Activity

Prior to your practical session for **Worksheet 17**, you are required to do the following:

1. Read through the instructions and questions in Practical Worksheet 17.
2. View the videos below.

Please submit this pre-lab worksheet when you report for your practical session.

Tests	Brief Description of Video	Shortened URL	QR Code
1	Chemical test using 2,4-dinitrophenylhydrazine	https://youtu.be/yo1ygeTUbO8	
2	Chemical test using Tollens' reagent (includes preparation of Tollens' reagent)	https://goo.gl/WNKs85	
3	Chemical test using Fehling's solution	http://tinyurl.com/Fehling-solution	
4	Chemical test using acidified KMnO ₄ (aq)	https://goo.gl/ta8aqS	
5	Chemical test using alkaline I ₂ (aq)	https://goo.gl/kAxGIQ	

Guide to Organic Qualitative Analysis

Chemical Test	Observations	Possible functional groups/structure present
Addition of aqueous bromine	Orange aqueous bromine decolourises.	<ul style="list-style-type: none"> • Alkene, • Phenol or • Phenylamine
KMnO ₄ , dilute H ₂ SO ₄ with heating in water bath	Purple KMnO ₄ decolourises.	<ul style="list-style-type: none"> • Primary /secondary alcohol • Aldehyde • Alky arene (Side chain oxidation) • Alkene (oxidative cleavage)
Addition of 2,4-dinitrophenylhydrazine (2,4-DNPH)	Orange ppt formed.	<ul style="list-style-type: none"> • Aldehyde • Ketone
Aqueous Iodine, NaOH(aq), heat	Yellow ppt formed.	<ul style="list-style-type: none"> • Presence of CH₃CH(OH)- • Presence of CH₃CO- group . <p>Note: Combine with reaction with 2,4-DNPH to determine if functional group is an alcohol or carbonyl.</p>
Tollens' reagent (silver diammine solution)	Silver mirror formed	<ul style="list-style-type: none"> • Aldehyde present. (R-CHO)
Fehling's solution (alkaline solution of Cu(II) complex ion)	Red precipitate of Cu ₂ O formed	<ul style="list-style-type: none"> • Aliphatic aldehyde present. (Cannot be benzaldehyde)

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Experiment 17: Organic Analysis 2 (alcohols and carbonyl group of ketone and aldehyde)

Answer **ALL** the questions in the SPACES provided.

- (a) Half fill a 250 cm³ beaker with water and heat until the water is almost boiling. This is a hot water bath for use later in the question. Turn off the Bunsen burner.

You should also prepare Tollens' reagent. Follow the instructions in the box below.

To 3 cm depth of aqueous silver nitrate in a test-tube, add 6 drops of aqueous sodium hydroxide. Then add aqueous ammonia dropwise, with shaking, until the brown solid **just** dissolves.

Do not add an excess of aqueous ammonia.

- (i) Carry out the following tests on **FA 5**, **FA 6** and **FA 7**. They are all organic liquids that contain carbon, hydrogen and oxygen only. Observe carefully, recording any changes seen. If it appears that no reaction has taken place, this should be clearly recorded.

Some tests have already been carried out and the observations recorded in the table.

FA 5 -propanal; FA 6 -ethanol; FA 7 -propanone

test	observations		
	FA 5	FA 6	FA 7
To 1 cm depth of the organic liquid in a test-tube, add a few drops of 2,4-dinitrophenylhydrazine. Leave the test-tube to stand in the hot water bath.	Orange ppt.	No orange ppt.	Orange ppt. [1]
To 1 cm depth of Tollens' reagent in a test-tube, add three drops of the organic liquid. Leave the test-tube to stand in the hot water bath.	Silver mirror	No silver mirror	No silver mirror [1]
To 1 cm depth of the organic liquid in a test-tube, add 1 cm depth of dilute sulfuric acid, followed by five drops of potassium manganate(VII) solution. Leave the test-tube to stand in the hot water bath.	Purple KMnO ₄ decolourised when warmed	Purple KMnO ₄ decolourised when warmed	Purple KMnO ₄ not decolourised [1] when warmed
To 1 cm depth of aqueous sodium hydroxide in a test-tube, add 1 cm depth of aqueous iodine. Then add three drops of the organic liquid.	No pale yellow ppt.	pale yellow precipitate	Pale yellow ppt. [1]

[4]

- (ii) From the observations in (a)(i), identify the functional group present in each organic liquid.

FA 5 **Aldehyde [1]**

FA 6 **Primary or secondary alcohol [1]**

FA 7 **Ketone [1]** [3]

- (iii) Use the observations to identify a structural feature that is present in **FA 6** and **FA 7**.

FA 6 **Contains $\text{CH}_3\text{CH}(\text{OH})-$ group [1]**

FA 7 **Contains $\text{CH}_3\text{CO}-$ group [1]** [2]

[CONTINUED NEXT PAGE]

(b) Planning

The labels of three bottles have fallen off. Each bottle contains **one** of the following:

- butan-2-ol
- butanal
- butanone

- (i)** Describe the procedures of simple chemical tests by which you could identify each of the organic compounds. You are **not** to identify the compounds by elimination.

Test 1: To identify butanal from butan-2-ol and butanone.

In separate test-tubes, place 1 cm depth of **Tollens' reagent**, followed by 3 drops of the unknown organic compounds. Heat the test-tubes in a hot water bath. [1]

The test-tube that gives a **silver mirror** contains **butanal**. [1] The two test-tubes with **no silver mirror** contain **butan-2-ol or butanone**. [1]

Test 2: To identify butanone from butan-2-ol.

In separate test-tubes, place 1 cm depth of each unknown organic compound, followed by a few drops of **2,4-dinitrophenylhydrazine**. Heat the test-tubes in a hot water bath. [1]

The test-tube that gives an **orange ppt** contains **butanone**. [1] The test-tube with **no orange ppt** contains **butan-2-ol**.

Test 3: To confirm identity of butan-2-ol.

Place 1 cm depth of aqueous NaOH in a test-tube and add 1 cm depth of aqueous iodine. Then add 3 drops of the unknown organic compound. Heat the test-tube in a hot water bath. [1]

A **pale yellow ppt** of CHI_3 formed confirms presence of **butan-2-ol**. [1]

Accept any other feasible tests.

[7]

- (ii)** For the tests you proposed in **(b)(i)**, state and briefly explain any **one** issue of reliability or limitations that may be present.

For **Test 2** using **2,4-dinitrophenylhydrazine**, butanal needs to be identified before butanone since both give an orange ppt, or vice-versa.

For **Test 3 (triiodomethane test)**, butanone needs to be identified before butan-2-ol since both give yellow ppt, or vice-versa. [2] any one with explanation

- (iii)** Suggest, with explanation, **two** safety measures to be taken for the tests proposed in **(b)(i)**.

Ensure no naked flame is used since all 3 organic compounds are flammable. [1] with reason

Perform experiment in a fume cupboard to minimise fumes inhaled since the organic compounds are volatile. [1] with reason. Accept any other relevant safety measures. [2]

[Total: 20]

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Practical Worksheet 18: Inorganic Analysis 2

Pre-Lab Activity

Prior to your practical session for **Worksheet 18**, you are required to do the following:

1. Read through the Chapter 7.2 of this Handbook on Inorganic Chemistry
2. Go through the thinking process

1. Preliminary Analysis of Unknown

- Colour of solution
- Colour of precipitate
 - Solubility in limited and excess amount of reagent to be recorded accordingly (if applicable)
- Gas evolved
 - Observations (any effervescence, smell, colour)
 - Describe test for gas
 - Give the identity of the gas

2. Tests to identify the unknown

Test	Things to note.
(i) To a 1 cm depth of FA 1 in a test-tube, add an equal depth of hydrogen peroxide .	<p>In this context, H_2O_2 decomposes because the metal ion present in FA1 acts as a catalyst. Write the equation for the decomposition of H_2O_2:</p> <p>.....$\text{H}_2\text{O}_2 \rightarrow \text{H}_2\text{O} + \frac{1}{2}\text{O}_2$.....</p> <p>From your equation, what gas should you prepare to test for?</p> <p>Test for the presence of O_2 gas.</p> <p>How should this test be carried out, and what is expected to be observed?</p> <p>Use of glowing splint and it should relight</p> <p>Note to students: Also observe for colour change in the solution.</p>
(ii) To a 1 cm depth of FA 1 in a test-tube, add an equal depth of aqueous sodium hydroxide, then (next page)	<p>Hint: Refer to pages 7-16 of this Practical Handbook. What is the purpose of NaOH(aq)?</p> <p>NaOH is a precipitating agent by supplying high concentration of OH^- as it is a strong base, fully ionised in solution ($\text{NaOH} \rightarrow \text{Na}^+ + \text{OH}^-$). So those insoluble hydroxides with low K_{sp} will be normally be precipitated.</p> <p>What observations should you look out for?</p> <p>Colour of precipitates formed and whether they are soluble in excess NaOH(aq)</p>

<p>Add a 1 cm depth of hydrogen peroxide.</p>	<p>In this context, H_2O_2 is acting as an oxidising agent. (H_2O_2 itself is <u>reduced</u>.)</p> <p>With reference to the data booklet, write the half equation of the reduction of H_2O_2.</p> $\text{H}_2\text{O}_2 + 2\text{H}^+ + 2\text{e}^- \rightarrow 2\text{H}_2\text{O}$ <p>From your equation above, is a gas expected to be evolved?</p> <p>No.</p> <p>Note to students: Also observe for colour changes. Effervescence may still be seen as the decomposition of H_2O_2 will also take place.</p>
<p>(iii) To a 1 cm depth of FA 1 in a boiling tube, add a 1 cm depth of aqueous sodium hydroxide. Heat the boiling tube gently.</p>	<p>Hint: Refer to pages 7-16 of this Practical Handbook.</p> <p>What are you testing for when NaOH(aq) is added followed by heating?</p> <p>Presence of ammonium ion, NH_4^+</p> <p>Note to students: Usually when there is gentle heating, prepare to test for gas.</p> <p>What gas is expected to be evolved?</p> <p>Ammonia gas</p> <p>How should this test be carried out, and what is expected to be observed?</p> <p>Use of moist red litmus paper and the red litmus paper turns blue.</p> <p>Why should the litmus paper not touch the mouth of the test tube?</p> <p>Some NaOH(aq) that was poured into the test tube could still be present at the mouth of the test tube. This will cause the red litmus paper to turn blue even without the evolution of ammonia gas.</p>
<p>(iv) To a 1 cm depth of FA 1 in a test-tube, add a few drops of aqueous barium nitrate.</p>	<p>What ion is being tested for when aqueous barium nitrate is added?</p> <p>Sulfate or sulfite.</p> <p>What is expected to be observed for a positive test?</p> <p>White precipitate</p> <p>Note to students: Observe for any change in solubility of the precipitate when excess HCl(aq) is added.</p>

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Experiment 18: Inorganic Analysis 2

Answer **ALL** the questions in the SPACES provided.

In this experiment, you will carry out tests, make observations and come to conclusions about the ions present in the compounds.

If the evolution of a gas is observed at any stage, the gas should be tested and identified. Details of the test carried out, the observations from the test and the identity of the gas should be given with the observations.

If it appears that no reaction has taken place, this should be clearly recorded.

- (a) **FA 1** is an aqueous solution that contains two cations and one anion from the list given in the *Qualitative Analysis Notes*.

Carry out the following tests on **FA 1** and complete the table to show your observations.

tests	observations
(i) To a 1 cm depth of FA 1 in a test-tube, add an equal depth of hydrogen peroxide.	<p>pale green solution turns orange-yellow. [1]</p> <ul style="list-style-type: none"> • effervescence; • gas evolved relights a glowing splint. • O₂(g) evolved. <p>Note: Students tend to fall short in the complete description of the gas evolved.</p>
(ii) To a 1 cm depth of FA 1 in a test-tube, add an equal depth of aqueous sodium hydroxide, then	<ul style="list-style-type: none"> • dirty-green / green ppt. • insoluble in excess NaOH. <p>Note: Students should realise that Fe²⁺ is present.</p>
add a 1 cm depth of hydrogen peroxide.	<p>ppt. turns reddish-brown; [1] (Fe(OH)₃ precipitated.)</p>

(H₂O₂ oxidised Fe²⁺ to Fe³⁺ as mentioned in pre-lab) Note: Students should confirm that Fe²⁺ was present in the FA1.

(iii) To a 1 cm depth of FA 1 in a boiling tube, add a 1 cm depth of aqueous sodium hydroxide. Heat the boiling tube gently.	<p>dirty-green /green ppt.</p> <p>pungent gas evolved; Gas turns moist red litmus paper blue.</p> <p>NH₃(g) evolved.</p> <p>(NH₄⁺ present)</p>
(iv) To a 1 cm depth of FA 1 in a test-tube, add a few drops of aqueous barium nitrate.	<p>white ppt. [1]</p> <p>(BaSO₄ or BaSO₃ precipitated) (SO₄²⁻ or SO₃²⁻ present)</p>

From your observations, identify the ions present in **FA 1**.

The cations are **Fe²⁺** and **NH₄⁺**

The anion is **SO₄²⁻ or SO₃²⁻** [1] for all

[7]

(b) **FA 2** is an aqueous solution that contains the **same** anion as that in **FA 1**.

(i) To a 2 cm depth of **FA 2** in a test-tube, slowly add a **small** spatula of solid sodium carbonate.

What did you observe?

brisk effervescence; gas evolved forms white ppt with limewater

∴ CO₂(g) liberated. NOTE: Students tend to forget to identify the gas.

From your observations, identify the cation present in **FA 2**.

H⁺ [2]

(ii) What other test could you carry out on **FA 2** to confirm the identity of the cation?
You should include the test and the results expected.

Do not carry out this test.

To 1 cm depth of FA 2 in a test-tube, add a small piece of magnesium ribbon.

Effervescence observed to form; gas evolved gives a "pop" sound with a

lighted splint. H₂(g) liberated.

[1]

[1] any relevant test and expected results

(c) Planning

Suppose you are given a solution containing Zn^{2+} , Al^{3+} , Cl^- and I^- ions, labelled **FA 3**.

- (i) From the *Qualitative Analysis Notes*, select two reagents, when used together, would allow you to separate the two anions in **FA 3**.

reagents **AgNO₃(aq)** and **NH₃(aq)** [1]
[1]

Students, for clarity in your answers, write AQUEOUS for both.

- (ii) Using the reagents you have identified in (c)(i), you are to devise a sequence of steps, using test-tube reactions, which would allow you to separate the two anions in **FA 3** so that each anion is present in a separate precipitate.

In your plan, you should include details on the reagents you would use, expected observations and the location of each anion (i.e. in a solution or precipitate) after each step.

Complete the table below and draw a line across the table after each numbered step.

Do not carry out your plan.

proposed step	expected observations	location of each anion
<u>1. Add AgNO₃(aq) until in excess.</u> (This is to ensure maximum amount of ppt formed.) <u>Filter</u> the mixture. Discard the filtrate.	Yellow ppt [white ppt is obscured by the yellow ppt. It CANNOT be seen. Whoever wrote white ppt will not get credit.]	Both I⁻ and Cl⁻ are in ppt (as AgX) [1 for both with observation]
<u>2. Add excess NH₃(aq) to the residue.</u> <u>Filter</u> the mixture.	Yellow ppt (residue) Colourless filtrate [1] for both	I⁻ in ppt (residue). Cl⁻ in solution (filtrate) [1] for both
<u>3. Add HNO₃(aq) to the filtrate [1]</u> (H ₂ SO ₄ cannot be used because it will react with Ag ⁺ to form a white ppt of Ag ₂ SO ₄ .)	White ppt	Cl⁻ in ppt (as AgCl) [1 with observation]

[5]

- (iii) From the *Qualitative Analysis Notes*, select reagent(s) which would allow you to separate the two cations in FA 3 so that **each cation is present in a separate precipitate. (STUDENTS DON'T SEEM TO NOTICE THIS INSTRUCTION!)**

In the table below, give a description of your proposed sequence of steps, the expected observations and the location of each cation (i.e. in a solution or precipitate) after each step.

Draw a line across the table after each numbered step.

Do not carry out your plan.

<i>proposed step</i>	<i>expected observations</i>	<i>location of each cation</i>
1. Add <u>excess NH₃(aq)</u> . <u>Filter the mixture [1]</u>	White ppt (residue) Colourless filtrate	Al ³⁺ in ppt (residue) Zn ²⁺ in solution (filtrate) [1 for both with observation]
2. Add <u>HNO₃(aq) (or H₂SO₄) dropwise to the filtrate [1]</u> Be careful not to use excess acid or the ppt will dissolve again.)	White ppt	Zn ²⁺ in ppt [1 with observation]

[4]

[Total: 20]

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Practical Worksheet 19: Inorganic Analysis 3

Pre-Lab Activity

Prior to your practical session for **Worksheet 19**, you are required to do the following:

3. Read through Chapter 7.2 of this Handbook on Inorganic Chemistry
1. on Inorganic Chemistry
2. Go through the thinking process

3. Tests to identify the unknown

Test	Things to note.															
This part is relevant to experiment 19 (b)(i)	<p>What observations should you look out for?</p> <p>Colour of precipitates, solubility in limited and excess NaOH(aq)</p> <p>.....</p>															
(i) To 1 cm depth of the filtrate in a test-tube, add aqueous sodium hydroxide, then	<p>Which precipitates obtained will change colour upon contact with air?</p> <p>Mn(OH)₂ and Fe(OH)₂</p> <p>.....</p>															
add aqueous hydrogen peroxide.	<p>H₂O₂ can act as both an oxidising and reducing agent.</p> <p>In the presence of an oxidising agent, H₂O₂ is oxidised to oxygen gas</p> <p>In the presence of a reducing agent, H₂O₂ is reduced to water</p> <p>If effervescence is observed, describe the test for the gas evolved.</p> <p>Place a glowing splint into the test tube. If oxygen gas is evolved, the gas should rekindle the glowing splint.</p> <p>.....</p>															
This part is relevant to experiment 19 (b). Read Page 7-15 of Handbook and watch the video on How to Make Fluted Filter Paper  https://youtu.be/caXpfoVqqXo	<p>Action of dilute acids on an unknown substance</p> <table border="1"> <thead> <tr> <th>Nature of unknown</th> <th>To test for</th> <th>To look out for</th> <th>Remarks</th> </tr> </thead> <tbody> <tr> <td>solid/solution</td> <td>anion</td> <td>gas</td> <td> <ul style="list-style-type: none"> • If carbon dioxide was evolved, carbonate is present • If sulfur dioxide was evolved, sulfite (SO_3^{2-}) is present • If nitrogen dioxide was evolved, nitrite (NO_2^-) is present </td> </tr> <tr> <td>solid</td> <td>metal</td> <td>gas</td> <td> <ul style="list-style-type: none"> • If hydrogen gas was evolved, the unknown is a metal. </td> </tr> </tbody> </table>				Nature of unknown	To test for	To look out for	Remarks	solid/solution	anion	gas	<ul style="list-style-type: none"> • If carbon dioxide was evolved, carbonate is present • If sulfur dioxide was evolved, sulfite (SO_3^{2-}) is present • If nitrogen dioxide was evolved, nitrite (NO_2^-) is present 	solid	metal	gas	<ul style="list-style-type: none"> • If hydrogen gas was evolved, the unknown is a metal.
Nature of unknown	To test for	To look out for	Remarks													
solid/solution	anion	gas	<ul style="list-style-type: none"> • If carbon dioxide was evolved, carbonate is present • If sulfur dioxide was evolved, sulfite (SO_3^{2-}) is present • If nitrogen dioxide was evolved, nitrite (NO_2^-) is present 													
solid	metal	gas	<ul style="list-style-type: none"> • If hydrogen gas was evolved, the unknown is a metal. 													

This part is relevant to experiment 19 (c)
Read Page 7-16 of Handbook

Watch the following video on thermal decomposition of hydrated calcium nitrate.



<https://www.youtube.com/watch?v=VNTkJWkPbiM>

Always use CLEAN AND DRY boiling tubes (or test-tubes) for heating

- Heat gently at first, then strongly until *no further change*

Write an equation for the thermal decomposition of hydrated calcium nitrate, $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$.



State and explain whether magnesium nitrate would undergo thermal decomposition at a higher or lower temperature than calcium nitrate.

Magnesium nitrate would decompose at a lower temperature. Mg^{2+} has a higher charge density and thus polarises the N-O bond/nitrate ion to a larger extent and the bond is weaker. $\text{Mg}(\text{NO}_3)_2$ undergoes thermal decomposition more readily.

Why is it necessary to heat the sample gently first?

To prevent spattering of the sample.

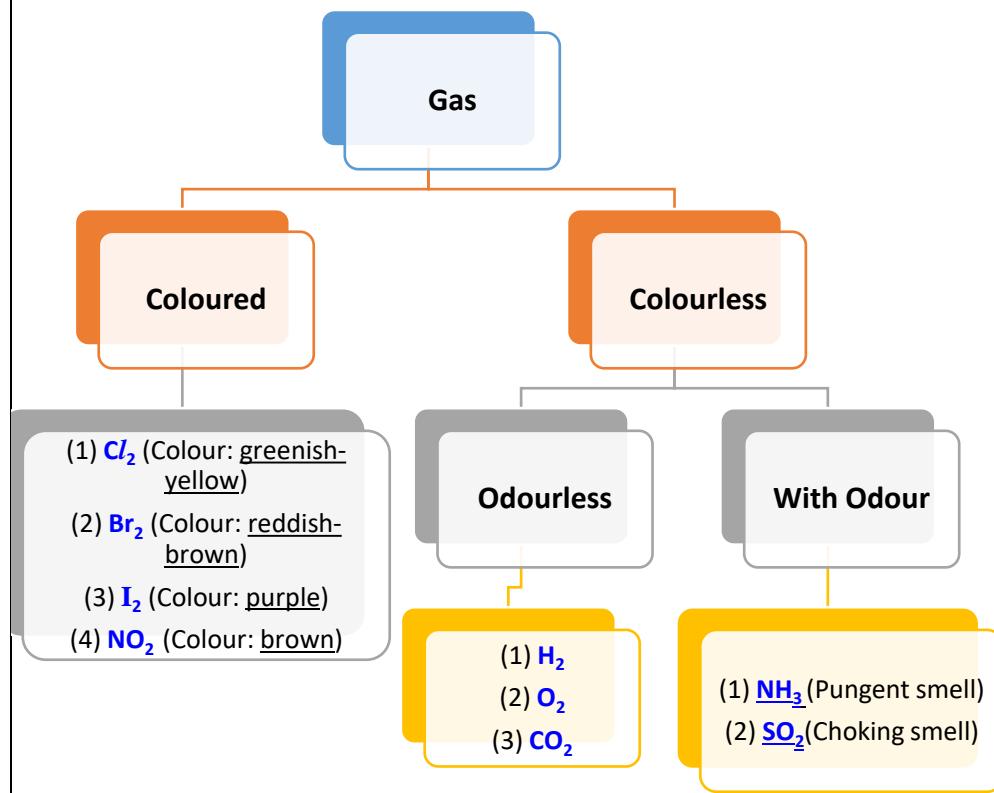
Why must the whole length of boiling tube be heated?

To prevent condensation of water vapour on cold parts of the test tube and subsequently cracking of boiling tube

NO_2 gas evolved is toxic and the fumes must not be inhaled. State a safety precaution that should be made.

The boiling tube containing NO_2 gas should be left in a fume cupboard.

The preliminary observation of the gas evolved is done before the test for gases is carried out. Complete the following flowchart with the gases that can be found in the Qualitative Analysis table.



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Experiment 19: Qualitative Analysis

Answer **ALL** the questions in the SPACES provided.

In this experiment, you will carry out tests, make observations and come to conclusions about the ions present in the compounds.

At each stage of any test, you are to record details of the following:

- colour changes seen;
- the formation of any precipitate; and
- the solubility of such precipitates in an excess of the reagent added.

Where gases are released, they should be identified by a test, **described in the appropriate place in your observations.**

(a) **FA 5, FA 6 and FA 7** are aqueous solutions each containing one cation and one anion.

(i) Carry out the following tests and record your observations for each solution in the table.

test	observation			[1] all 3 correct
	FA 5	FA 6	FA 7	
To 1 cm depth of the unknown solution in a test-tube, add 1 cm depth of aqueous sodium carbonate.	no reaction	effervescence; gas gives white ppt with limewater. $\text{CO}_2(\text{g}) \square$	effervescence; gas gives white ppt with limewater. $\text{CO}_2(\text{g}) \square$	
To 1 cm depth of the unknown solution in a test-tube, add 1 cm depth of aqueous copper(II) sulfate.	blue ppt	no reaction / no change / no ppt / pale blue solution <i>[not 'no observation']</i>	no reaction / no change / no ppt / pale blue solution <i>[not 'no observation']</i>	[1] all 3 correct
To 1 cm depth of the unknown solution in a test-tube, add 1 cm depth of aqueous barium chloride or aqueous barium nitrate.	white ppt	white ppt	no ppt	[1] all 3 correct

[3]

(ii) What ion is present in both **FA 6** and **FA 7**? [1]

H^+ [1]

(iii) The anion in **FA 5** is one of carbonate, chloride, nitrate or sulfate.

Which anion is present in **FA 5**? [1]

CO_3^{2-} [1]

(iv) Write an ionic equation, including state symbols, for the reaction of **FA 5** with aqueous copper(II) sulfate.



[1]

- (b) FA 8 contains two cations and two anions from the list given in the *Qualitative Analysis Notes*.

- To 5 cm depth of distilled water in a boiling tube, add a heaped spatula (small) of FA 8.
- Shake the boiling tube thoroughly for one minute to make sure that no more of the solid will dissolve.
- Filter the mixture into a clean boiling tube.
- Place the filter funnel in a conical flask and wash the residue with a little distilled water.
- **Keep both filtrate and residue for tests (i) and (ii) below.**

(i) Tests on the filtrate.

Carry out the following tests and record your observations in the table below.

test	observations
To 1 cm depth of the filtrate in a test-tube, add aqueous sodium hydroxide, then	off-white ppt rapidly turning brown on contact with air; insoluble in excess NaOH [1] <i>not cream ppt</i>
add aqueous hydrogen peroxide.	ppt. turns darker brown/brown-black [1] brisk effervescence; gas <i>relights a glowing splint</i>; O₂(g) liberated [1]

[3]

(ii) Tests on the residue.

Carry out the following tests and record your observations in the table below.

test	observations
Place the funnel containing the residue into a clean boiling tube. Pour approximately 5 cm ³ of dilute nitric acid onto the residue. Collect 1 cm depth of solution in the boiling tube. Remove the funnel and return it to the conical flask.	effervescence; gas gives white ppt with limewater. CO₂(g) liberated. [1] colourless solution
To this solution in the boiling tube, add aqueous sodium hydroxide.	white ppt; insoluble in excess NaOH [1]

[2]

- (iii) The **two** cations present in FA 8 areand..... **Mn²⁺** **Mg²⁺** [1 for both] [1]

- (iv) One anion present in FA 8 is **CO₃²⁻** [1] [1]

- (v) Suggest what type of reaction takes place when hydrogen peroxide is added in test (b)(i).

redox [1] accept oxidation of Mn [1]

(c) Solid FA 9 contains one cation and one anion from the list given in the *Qualitative Analysis Notes*.

- (i) Place approximately half of your FA 9 in a test-ube and heat. Record your observations and identify two gases that are formed.

observations
colourless crystal melts to give a colourless liquid; crackling sound;
water droplets condensed on cooler part of test-tube; brown gas evolved
and another colourless gas that relights a glowing splint; yellow residue

} [1] 2 gases
+ another 2 observations

gases formed NO₂ (g) and O₂ (g) [1 for both]

[2]

- (ii) Dissolve the remaining FA 9 in 1 cm depth of deionised water in a test-tube.
 Divide this solution into two portions

- To the first portion, add aqueous sodium hydroxide dropwise, until in excess.

observations
white ppt;

ppt dissolves in excess NaOH to give a colourless solution [1 for both]

From these observations, cations that could be present in FA 9 are Al³⁺ and Zn²⁺ [1]

- Choose a reagent that would allow you to distinguish between these possible cations.

reagent aqueous NH₃

Use this reagent and the second portion of FA 9 to identify the cation present.

observations
white ppt;

ppt dissolves in excess NH₃ to give a colourless solution [1 for both]

cation present in FA 9 is Zn²⁺ [1]

[4]

[Total: 20]