RAFFLES INSTITUTION RAFFLES PROGRAMME - YEAR THREE CHEMISTRY

EXPERIMENTAL DESIGN AND PURIFICATION TECHNIQUES

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Preparation of Gas	Specific Test	Observations
<u>Chlorine</u> Put a spoonful of manganese (IV) oxide, MnO_2, in a dry test tube. Add $1cm^3$ of hydrochloric acid. Warm the mixture.	Test gas evolved with moist blue litmus paper.	Pale green/yellow gas evolved. The blue litmus paper turns red and then white/is bleached.
<u>Hydrogen</u> To 5 pellets of Zinc in a test tube, add dilute sulfuric acid until half full. Add 2 drops of aqueous copper (II) sulfate.	Insert a **lighted** wooden splint slowly into the test tube.	Effervescence observed. Colorless gas evolved. Neither blue nor red litmus paper changes color. The gas extinguishes the lighted splint with a "pop" sound. A red-brown solid is formed.
<u>Oxygen</u> Add ¼ spoon of manganese (IV) oxide solid into a dry test tube. Using a dropper add about $1cm^3$ of aqueous hydrogen peroxide, H_2O_2	Insert a **glowing** splint into the test tube.	Effervescence observed. Colorless gas evolved. Neither blue nor red litmus paper changes color. The gas rekindles/relights/reignites the glowing splint.
<u>Sulfur Dioxide</u> Put a spoonful of sodium sulfite, Na_2SO_3, in a test tube. Pour in dilute hydrochloric acid until half full. Warm gently.	Dip a piece of filter paper strip into acidified potassium manganate (VII). Hold the filter paper strip at the mouth of the test tube.	Colorless and choking gas evolved. Gas turns moist blue litmus paper red. The purple potassium manganate (VII) becomes colorless
<u>Carbon Dioxide</u> Add a few pieces of calcium carbonate (marble chips) into a test tube. Pour in hydrochloric acid or dilute nitric acid until half full.	Pass the gas through limewater (Calcium hydroxide Ca(OH)2). (Use a delivery tube or dropper and about $2cm^3$ limewater in a test tube)	Effervescence observed. Colorless gas evolved. Gas turns moist blue litmus paper red. A white precipitate is formed in the limewater.
<u>Ammonia</u> Add a spoonful of ammonium chloride solid into a test tube. Pour in sodium hydroxide solution until about ⅓ full. Warm the mixture.	Test the gas evolved with a piece of moist red litmus paper.	Colorless and pungent gas evolved. Moist red litmus turns blue. Effervescence evolved.

Measuring volume of gas: Gas syringe

Collecting gas depends on

- Solubility in water: displacement of water (for insoluble gases)
- Density compared to that of air: downward delivery or upward delivery
- Drying agent when gas produced is contaminated with water vapour
    - o Concentrated sulfuric acid (should not be used in conjunction with a base as they will react)
    - o Anhydrous calcium chloride
    - o Anhydrous copper (II) sulfate (white in colour)
    - o Anhydrous calcium oxide (should not be used in conjunction with an acid)
    - o Silica gel
    - o Indicator: Anhydrous copper (II) sulfate (turns from white to blue in the presence of water)

Measuring volume of liquid:

- Measuring cylinder
- Pipette: 1 d.p.
- Burette: 2 d.p.

Experimental Design

Measuring Mass: Electronic balance

Filtering - Suspension

1. Filter the mixture and collect all the filtrate/residue
2. Wash the residue with distilled water
3. Dry the residue in a low-heat oven (if mass is going to be measured) / pressing between two pieces of filter paper (if mass does not matter and only product is required)

Crystallization - Salt decomposes at high heat

1. Heat the solution in an evaporating dish.
2. When about one-third of the solution is left stop heating – this produces a hot saturated solution.
3. Let the saturated solution cool and crystals would appear.
4. Filter to remove the crystals from remaining solution.
5. Dry the crystals by pressing them between sheets of filter paper.

Sublimation

1. Heat the mixture in an evaporating dish and invert a filter funnel over the mixture.
2. The solid sublimes, and its vapour solidifies as a deposit on the cooler inner surface of the inverted funnel.

The solids that sublime are: ammonium chloride, iodine, dry ice (solid carbon dioxide) and naphthalene (mothball).

Separating Funnel - Immiscible liquids

Simple Distillation: Recover solvent from solution

1. Boil solution together with some broken porcelain in a flask that is connected to a condenser.
2. Pump cooling water into the jacket of the condenser.
3. The vapour in the flask enters the condenser and is condensed back into liquid, which is collected in the conical flask as the distillate.

What is the purpose of the broken porcelain?

They act as boiling chips or anti-bumping granules, which ensures smooth boiling by allowing formation of smaller air bubbles.

Why is the thermometer bulb placed at the exit of the flask?

This allows the thermometer to measure the temperature of the pure vapour exiting the flask, thus determining the boiling point of the solvent) in the mixture.

Why does the cooling water enter the bottom of the condenser rather than the top?

This ensures that the bottom of the condenser is the coolest part, and any vapour that did not condense initially would condense here, thus no vapour can escape.

Fractional Distillation: Separate a mixture of miscible liquids with different boiling points

1. Put the mixture in a flask that is linked to a fractionating column, which is in turn linked to a condenser.
2. Heat the mixture with an electric heater.
3. The heat vaporizes the liquids and drives the vapours up the fractionating column. (…) has a higher boiling point than (…) , thus the (…) vapour condenses in the fractionating column and drips back into the flask. The (…) vapour reaches the top of the column and passes into the condenser and is thus condensed, forming (…) liquid as the distillate.

Why are tiny glass beads used to fill the fractionating column?

This increases the surface area for the condensation of vapour making separation of the two vapours more efficient.

Reverse osmosis: Purifying a solvent

Process of forcing a solvent from a region of high solute concentration through a partially permeable membrane to a region of low solute concentration by applying pressure in excess of osmotic pressure.

Important skills in planning

1. Give theoretical basis of why a certain method is used to solve the equation.
2. List the sequence of manipulations proposed in the procedure in a coherent sequence.
3. Provide appropriate details regarding apparatus and materials used.
4. State safety precautions and give reasons why these precautions are taken.
5. State the measures taken to ensure reliability of results and how these measures ensure reliability.
6. Represent apparatus using labeled scientific diagrams.

Question-answering techniques

- Be specific in all explanations
- Use information provided in the question
- Precision must be accurate (exact smallest division for length and angle; half smallest division for all else)