Edexcel International A-Level Chemistry: A Comprehensive Revision Guide for Unit 6 Practical Skills

1.0 Introduction: Mastering the Practical Skills Paper

Welcome to this comprehensive guide for the Edexcel International A-Level Chemistry Unit 6 paper. It is essential to approach this examination not as a test of your performance in a laboratory, but as a rigorous assessment of your *understanding* of the principles behind experimental chemistry. This paper evaluates your ability to design, execute, analyse, and critically evaluate chemical investigations. This guide will systematically deconstruct the required skills, techniques, and theoretical knowledge, providing a clear framework to build both your confidence and proficiency.

We will begin by establishing a strong foundation, reviewing the core laboratory techniques and apparatus that form the building blocks of all practical work. From there, we will progress to the specific applications of these techniques in qualitative and quantitative analysis, covering the identification of unknown substances and the precise measurement of chemical properties. Finally, we will consolidate this knowledge with key organic and inorganic preparations and conclude with invaluable examiner insights and exam strategies to help you avoid common pitfalls and maximise your performance.

2.0 Foundational Laboratory Techniques and Apparatus

Mastering the core laboratory techniques is of paramount strategic importance for success in Unit 6. These procedures are the fundamental building blocks for every experiment you will encounter. A thorough understanding of their purpose, correct execution, and inherent limitations is crucial for designing sound experimental methods and, ultimately, for earning marks. Each step in a procedure has a specific scientific justification, and recognising this is key to demonstrating a higher level of chemical understanding.

2.1 Heating Under Reflux

Heating under reflux is a technique used to heat a reaction mixture for an extended period without losing volatile reactants or products to evaporation.

- **Principle:** The apparatus allows for continuous boiling and condensation. Vapours produced from the boiling liquid rise into a vertical condenser, are cooled back into a liquid, and drip back down into the reaction flask. This allows the reaction to be carried out at the solvent's boiling point for a prolonged time, increasing the rate of reaction and enabling it to go to completion.
- **Apparatus Setup:**

- A pear-shaped or round-bottomed flask is used to contain the reaction mixture.
- A Liebig condenser is attached vertically to the neck of the flask.
- Cooling water enters the condenser jacket at the bottom inlet and exits at the top outlet to ensure the jacket remains full and provides the most efficient cooling.
- **Why this step is important:** The top of the condenser must **never** be sealed. Sealing the system would cause a dangerous build-up of pressure from the vaporising liquid, potentially causing the apparatus to explode.

- **Key Considerations:**
  - **Anti-bumping granules** are added to the flask before heating.
  - **Why this step is important:** These small, porous ceramic pieces promote smooth, even boiling by providing a surface for bubbles to form on, preventing sudden, violent boiling (known as flash boiling or bumping).

2.2 Distillation (Simple, Fractional, and Steam)

Distillation is a set of techniques used to separate and purify liquids based on differences in their boiling points. The choice of method depends on the nature of the mixture being separated.

Distillation Type	Primary Use	Key Apparatus Features
Simple Distillation	Separating a pure liquid from a solution, or separating liquids with significantly different boiling points (e.g., >25 °C difference).	A round-bottomed flask is heated, and the vapour passes into a condenser angled downwards. A thermometer is placed with its bulb at the level of the side-arm leading to the condenser.
Fractional Distillation	Separating a mixture of liquids with similar boiling points (e.g., <25 °C difference), such as separating crude oil into fractions.	Includes a fractionating column (packed with glass beads or rings) placed between the flask and the condenser. This provides a large surface area for repeated vaporisation and condensation cycles, enriching the vapour with the more volatile component.

Steam Distillation	Separating temperature-sensitive organic compounds or liquids that are immiscible with water. The substance distils at a temperature below its normal boiling point.	Steam is generated externally and bubbled through the reaction mixture. The mixed vapours of water and the organic compound are then passed through a condenser and collected.

- **Critical Placement:** For both simple and fractional distillation, the thermometer bulb must be positioned precisely at the T-junction leading to the condenser.
    - **Why this step is important:** This ensures that the temperature being recorded is the boiling point of the substance that is actually distilling into the condenser, providing an accurate measure of purity.
- **Heating Flammable Liquids:** When distilling flammable organic liquids, a water bath or an electric heating mantle should be used instead of a direct Bunsen flame.
    - **Why this step is important:** A naked flame presents a significant fire hazard and could ignite the flammable vapours.

2.3 Separation and Extraction

This technique is used to separate immiscible liquids, most commonly an organic layer from an aqueous layer, using a separating funnel.

- **Procedure:**
    1. Pour the mixture into the separating funnel, ensuring the tap is closed.
    2. Stopper the funnel, invert it, and shake gently to mix the layers.
    3. Periodically, open the tap while the funnel is inverted to release any pressure that has built up, for example, from the production of CO_2 gas during an acid neutralisation.
    4. Clamp the funnel and allow the layers to fully separate.
    5. Remove the stopper, then open the tap to carefully run off the lower layer into a beaker.
- **Washing the Organic Layer:** The organic product is often washed to remove impurities. For instance, washing with sodium hydrocarbongate solution neutralises any remaining acid catalyst.

    - **Why this step is important:** To confirm that all the acid has been neutralised, the aqueous layer can be tested with damp red litmus paper, which should turn blue.

- **Improving Separation:** Sometimes, adding saturated sodium chloride solution to the funnel can improve the separation.
    - **Why this step is important:** This increases the density of the aqueous layer, making the boundary between the two layers sharper and more distinct.

2.4 Drying a Liquid Product

After separation, the organic layer will still contain traces of dissolved water. This water must be removed using a drying agent.

- **Purpose:** To remove residual water from an organic liquid.
- **Suitable Drying Agents:** Anhydrous salts such as **anhydrous calcium chloride**, **anhydrous magnesium sulfate**, or **anhydrous sodium sulfate** are commonly used. The agent must not react with the organic product.
- **Procedure:** Add the drying agent to the organic liquid and swirl.
    - **Why this step is important:** A sufficient amount of drying agent has been added when the liquid turns from cloudy to clear. The salt will often clump together as it absorbs water, and free-flowing powder indicates the liquid is dry. The dry liquid can then be decanted or filtered away from the solid drying agent.

2.5 Purification of a Solid: Recrystallisation

Recrystallisation is the primary method for purifying an impure solid product. The principle relies on the different solubilities of the desired compound and the impurities in a chosen solvent.

1. **Dissolve the impure solid in a minimum volume of a hot solvent.**
    - **Why this step is important:** Using the minimum volume ensures that the hot solution is saturated. This is critical for maximising the yield of pure crystals when the solution is cooled.
2. **Perform a hot filtration using a pre-heated, stemless funnel.**
    - **Why this step is important:** This step removes any *insoluble* impurities. The apparatus must be kept hot to prevent the desired product from crystallising

prematurely on the filter paper or in the funnel stem, which would reduce the final yield.

3. **Cool the filtrate slowly to room temperature, then in an ice bath.**
    - **Why this step is important:** As the solution cools, the solubility of the desired compound decreases, causing it to crystallise out. The *soluble* impurities remain dissolved in the cold solvent because they are present in much smaller quantities and the solution is not saturated with respect to them.
4. **Filter the pure crystals using a Büchner funnel under reduced pressure (vacuum filtration).**
    - **Why this step is important:** This method is much faster than gravity filtration and pulls more of the solvent through, resulting in a drier solid product.
5. **Wash the crystals with a small amount of cold solvent and dry them.**
    - The crystals are washed to remove any remaining dissolved impurities. The solvent must be cold to minimise loss of the purified product. Crystals can be dried between sheets of filter paper or in a desiccator.
    - **Why this step is important:** Drying in a desiccator is often preferred over using an oven, as heating could cause the product to melt or decompose, especially if it has a low melting point.

2.6 Purity Determination: Melting Point Analysis

The melting point of a solid is a physical property that provides a reliable indication of its purity.

- **Procedure:** A small sample of the dry solid is placed in a capillary tube, which is then heated slowly in a Thiele tube containing oil or in an electronic melting point apparatus. The temperature range from which the solid begins to melt until it has completely turned into a liquid is recorded.
- **Interpreting Results:**
    - A **pure substance** will have a sharp melting point (melting over a narrow range of 1-2 °C) that is consistent with the value found in data literature.
    - An **impure substance** will melt at a lower temperature and over a broad range. The impurities disrupt the regular crystal lattice structure, requiring less energy to break it down.

2.7 Standard Titration Procedure

Titration is a quantitative technique used to determine the concentration of a solution by reacting it with another solution of known concentration (a standard solution). Accuracy is paramount.

- **Best Practices for Accuracy:**
    - **Apparatus Rinsing:** The burette must be rinsed with the solution it will contain (the titrant), and the pipette must be rinsed with the solution it will deliver (the analyte). The conical flask should only be rinsed with distilled water. Rinsing with the analyte solution would introduce extra, unmeasured moles of the substance, leading to an inaccurate, larger titre value.
    - **Burette Preparation:** The burette should be filled so the jet space below the tap is full and contains no air bubbles. An initial reading is taken.
    - **Accurate Readings:** All burette readings should be taken from the bottom of the meniscus at eye level to avoid parallax error. Readings should be recorded to two decimal places (e.g., 24.50 cm^3).
    - **Technique:** A white tile placed under the conical flask makes the colour change at the end-point easier to see. The flask should be swirled continuously, especially near the end-point, where the titrant should be added dropwise.
    - **Concordant Results:** A rough 'trial' titration is often performed first to find the approximate end-point. Titrations are then repeated until at least two results are concordant (within 0.20 cm^3 of each other). A mean titre is calculated using only these concordant results.
- **Common Acid-Base Indicators:**

Indicator	Colour in Acid	Colour in Base	Typical Use
Phenolphthalein	Colourless	Pink	Strong acid vs. Strong base; Weak acid vs. Strong base
Methyl Orange	Red	Yellow	Strong acid vs. Strong base; Strong acid vs. Weak base

Having mastered these foundational techniques, we can now explore how they are applied to identify unknown substances in qualitative analysis.

3.0 Qualitative Analysis: Identifying Inorganic and Organic Unknowns

Qualitative analysis is a core component of the Unit 6 assessment, requiring a logical and systematic approach to identify unknown substances through characteristic chemical reactions. Success in this area hinges on meticulous observation and a thorough knowledge of standard chemical tests. This section provides a comprehensive toolkit of these tests, organised to help you confidently identify a wide range of inorganic and organic compounds.

3.1 Tests for Inorganic Cations

Flame Tests

Flame tests are used to identify certain metal cations by the characteristic colour they impart to a Bunsen flame.

Cation	Procedure	Observed Flame Colour
Li^+	Dip a clean nichrome/platinum wire into concentrated HCl, then into the solid sample. Place the wire into a non-luminous Bunsen flame.	Scarlet / Red
Na^+	"	Yellow / Orange
K^+	"	Lilac
Ca^{2+}	"	Orange-red / Brick-red
Sr^{2+}	"	Crimson Red
Ba^{2+}	"	Green / Apple-green

Note: The wire is cleaned with concentrated HCl to remove contaminants. A non-luminous (blue) flame is used so that its colour does not mask the colour produced by the cation.

Tests with Sodium Hydroxide (NaOH) and Ammonia (NH_3)

Adding aqueous NaOH or NH_3 to solutions containing metal cations often produces insoluble metal hydroxide precipitates with distinctive colours and properties.

Cation	Initial Observation with $NaOH/NH_3$	Observation in Excess NaOH	Observation in Excess NH_3
Cu^{2+}	Blue precipitate	Insoluble	Precipitate dissolves to form a deep blue solution
Fe^{2+}	Green precipitate	Insoluble	Insoluble (precipitate turns brown on standing in air)
Fe^{3+}	Brown precipitate	Insoluble	Insoluble
Co^{2+}	Blue precipitate (turns pink on standing)	Insoluble	Precipitate dissolves to form a light brown solution
Ni^{2+}	Green precipitate	Insoluble	Precipitate dissolves to form a blue solution
Mn^{2+}	Off-white precipitate (turns brown on standing in air)	Insoluble	Insoluble
NH_4^+	*On warming with NaOH:* Pungent gas (ammonia) produced, turns damp red litmus paper blue	N/A	N/A
Mg^{2+}	White precipitate	Insoluble	Insoluble
Ca^{2+}	White precipitate (more soluble than $Mg(OH)_2$)	Insoluble	No precipitate
Zn^{2+}	White precipitate	Precipitate dissolves to form a colourless solution	Precipitate dissolves to form a colourless solution
Al^{3+}	White precipitate	Precipitate dissolves to form a colourless solution	Insoluble
Cr^{3+}	Green precipitate	Precipitate dissolves to form a green solution	Precipitate dissolves to form a violet solution

Note: The green precipitate of $Fe(OH)_2$ and the off-white precipitate of $Mn(OH)_2$ slowly oxidise in air to form brown precipitates.

3.2 Tests for Inorganic Anions

A series of simple chemical tests can be used to identify common anions in solution.

Anion	Reagent(s)	Positive Observation
Carbonate (CO_3^{2-})	Dilute acid (e.g., HCl), then bubble gas through limewater ($Ca(OH)_2$)	Effervescence (fizzing). The gas produced turns limewater milky/cloudy.
Sulfate (SO_4^{2-})	Dilute HCl followed by Barium chloride ($BaCl_2$) solution.	A dense white precipitate ($BaSO_4$) is formed. *Acid is added first to remove any carbonate ions, which would also form a white precipitate with Ba^{2+}.*
Halides (Cl^-, Br^-, I^-)	Dilute HNO_3 followed by Silver nitrate ($AgNO_3$) solution.	**Cl^-:** White precipitate (AgCl) *Soluble in dilute NH_3.* **Br^-:** Cream precipitate (AgBr) *Insoluble in dilute NH_3; soluble in concentrated NH_3.* **I^-:** Yellow precipitate (AgI) *Insoluble in both dilute and concentrated NH_3.*
Nitrate (NO_3^-)	Aqueous NaOH and aluminium foil, followed by gentle warming.	Bubbles of ammonia gas are produced, which turns damp red litmus paper blue.

3.3 Tests for Common Gases

Identifying the gas produced in a reaction is a key observational skill.

Gas	Test Procedure	Positive Observation
Hydrogen (H_2)	Place a lit splint into a test tube of the gas.	The splint extinguishes with a 'squeaky pop' sound.

Oxygen (O_2)	Place a glowing splint into a test tube of the gas.	The glowing splint relights.
Carbon dioxide (CO_2)	Bubble the gas through limewater (aqueous $Ca(OH)_2$).	The limewater turns milky/cloudy/chalky.
Ammonia (NH_3)	Hold damp red litmus paper near the gas.	The damp red litmus paper turns blue.
Chlorine (Cl_2)	Hold damp blue litmus paper near the gas.	The damp blue litmus paper is bleached (turns white).

3.4 Tests for Organic Functional Groups

These specific chemical tests produce distinct observable changes, allowing for the identification of key functional groups within an unknown organic molecule.

Functional Group	Test/Reagent	Conditions	Positive Observation
Alkene (C=C)	Bromine water (Br_2(aq))	Room Temperature	Orange solution turns colourless.
Haloalkane	Warm with aqueous Silver nitrate ($AgNO_3$)	Heat under reflux	A precipitate of the corresponding silver halide forms. **AgCl:** white; **AgBr:** cream; **AgI:** yellow. The rate of precipitation indicates the C-X bond strength: Iodoalkane (fastest) > Bromoalkane > Chloroalkane (slowest).
Alcohol (-OH)	Acidified Potassium Dichromate(VI) ($K_2Cr_2O_7/H^+$)	Heat under reflux	**Primary/Secondary:** Orange solution turns green. **Tertiary:** No change.
	Phosphorus pentachloride (PCl_5)	Room Temperature	Steamy white fumes of HCl gas.
Phenol	Iron(III) chloride ($FeCl_3$(aq))	Room Temperature	Purple coloration.

	Bromine water ($Br_2(aq)$)	Room Temperature	White precipitate forms.
Carbonyl (Aldehyde & Ketone)	2,4-Dinitrophenylhydrazine (2,4-DNPH)	Room Temperature	Orange/Yellow precipitate forms.
Aldehyde only	Tollen's Reagent ($[Ag(NH_3)_2]^+$)	Heat gently	A 'silver mirror' forms on the inside of the test tube.
	Fehling's/Benedict's Solution (Cu^{2+} complex)	Heat gently	Blue solution forms a brick-red precipitate (Cu_2O).
Carboxylic Acid (-COOH)	Sodium carbonate ($Na_2CO_3(aq)$)	Room Temperature	Effervescence (bubbles of CO_2 gas).
Methyl Ketone / Ethanal (the only aldehyde to give a positive result) / Ethanol	Iodoform Test (I_2 in NaOH(aq))	Warm gently	Pale yellow precipitate (CHI_3) with an antiseptic smell.

Having established how to identify substances, we now move to quantitative experiments, where precise measurement and calculation become the primary focus.

4.0 Quantitative Analysis: Core Experiments and Data Handling

Quantitative analysis shifts the focus from "what is it?" to "how much is there?" or "how fast is the reaction?". These experiments demand precision in measurement, proficiency in data manipulation, and an awareness of potential sources of error. Mastering these quantitative skills is essential for a complete understanding of experimental chemistry.

4.1 Following Reaction Rates (CP 9 & 10)

- **Titrimetric Method (Iodine-Propanone Reaction):** This experiment follows the concentration of a reactant over time. The reaction between iodine and propanone (catalysed by acid) is monitored by taking samples at regular intervals.
    - **Quenching:** Each sample is immediately added to sodium hydrogencarbonate solution. This neutralises the acid catalyst, effectively stopping the reaction at that specific time.

    - **Titration:** The amount of unreacted iodine in the quenched sample is then determined by titration with a standard solution of sodium thiosulfate.
    - **Rate Determination:** By keeping the propanone and acid in large excess, their concentrations remain effectively constant. This allows the order of reaction with respect to iodine to be determined from a graph of iodine concentration versus time.
- **Clock Reactions (Iodine Clock):** A clock reaction measures the time taken for a specific, observable change to occur. The rate is then taken as being proportional to 1/time. In the iodine clock reaction, a fixed amount of sodium thiosulfate is added to the reaction mixture. The thiosulfate immediately reacts with any iodine produced. Once all the thiosulfate is consumed, the next portion of iodine produced will turn a starch indicator blue-black, signalling the end-point.
- **Determining Activation Energy (Arrhenius Equation):** This experiment investigates how temperature affects the rate constant. The reaction between bromate, bromide, and phenol is carried out at several different temperatures, and the time taken for an indicator to be bleached is recorded.
    - The Arrhenius equation can be expressed in logarithmic form: $\ln(k) = -Ea/RT + \ln(A)$.
    - Since rate is proportional to 1/t, we can plot a graph of **ln(rate) versus 1/T** (where T is the absolute temperature in Kelvin).
    - This yields a straight line with a gradient equal to **-Ea/R**, where R is the gas constant (8.31 J K^{-1} mol^{-1}). This allows the activation energy (Ea) to be determined graphically. Note: Some practical instructions may ask you to plot ln(t) vs 1/T. Since rate $\propto$ 1/t, this graph would also be a straight line, but with a positive gradient equal to +Ea/R.

4.2 Enthalpy Change Determination (Calorimetry)

Calorimetry is used to measure the heat energy change in a chemical reaction.

- **Principle:** The heat change (q) is calculated using the formula **$q = mc\Delta T$**, where m is the mass of the solution being heated, c is the specific heat capacity of the solution (usually taken as that of water, 4.18 J g^{-1} K^{-1}), and ΔT is the temperature change.
- **Apparatus:** The reaction is carried out in an insulated container, such as a polystyrene cup placed inside a beaker, to minimise heat loss to the surroundings.

- **Finding an Accurate ΔT:** For reactions that are not instantaneous, heat is lost to the surroundings while the reaction is still proceeding. To account for this, temperature is recorded at regular intervals before, during, and after the reaction. A graph of temperature versus time is plotted. The cooling part of the curve is extrapolated back to the time of mixing, allowing for an accurate determination of the maximum theoretical temperature change.
- **Calculating Molar Enthalpy Change (ΔH):** The heat change q (in Joules) is divided by the number of moles of the limiting reactant to find the enthalpy change per mole. This value is then converted to kJ mol^{-1} and given the correct sign (negative for exothermic, positive for endothermic).
- **Common Sources of Error:**
    - Heat loss to the surroundings.
    - Assuming the specific heat capacity and density of the solution are the same as pure water.
    - Incomplete or slow reactions.
    - Ignoring the heat absorbed by the apparatus itself.

4.3 Investigating Electrochemical Cells (CP 12)

This practical involves constructing electrochemical cells and measuring their potential difference (E_cell).

- **Standard Setup:** An electrochemical cell consists of two half-cells. Each half-cell typically contains a metal electrode immersed in a 1.0 mol dm^{-3} solution of its own ions. The two half-cells are connected by:
    - A **high-resistance voltmeter** to measure the potential difference.
    - A **salt bridge** (e.g., filter paper soaked in saturated potassium nitrate, KNO_3), which completes the electrical circuit.
- **Function of the Salt Bridge:** The salt bridge allows ions to flow between the two half-cells to balance the charge build-up that occurs as the reaction proceeds, maintaining electrical neutrality.
- **Standard Conditions:** For a measured potential to be a *standard* electrode potential, specific conditions must be met: **298 K (25 °C)**, **1.0 mol dm^{-3}** concentration of all ions, and **100 kPa** pressure for any gases.

- **Discrepancies:** Experimental E_cell values often differ from theoretical data book values because the measurements were not taken under perfect standard conditions.

4.4 Redox Titrations (CP 13)

Redox titrations are based on oxidation-reduction reactions.

- **Iron(II) with Manganate(VII):**
    - **Procedure:** A solution containing Fe^{2+} ions is titrated with a standard solution of potassium manganate(VII) ($KMnO_4$).
    - **Indication:** No separate indicator is needed. $KMnO_4$ is **self-indicating**. The MnO_4^- ion is intense purple, while the Mn^{2+} ion it is reduced to is colourless. The end-point is the first permanent pale pink colour, indicating a slight excess of MnO_4^-.
    - **Acidification:** The reaction requires acidic conditions. Dilute sulfuric acid is the only suitable acid. **HCl cannot be used** because the MnO_4^- ion is a strong enough oxidising agent to oxidise Cl^- ions to Cl_2 gas. **HNO_3 cannot be used** as it is also an oxidising agent and would oxidise the Fe^{2+} ions itself.
- **Iodine with Sodium Thiosulfate:**
    - **Procedure:** A solution of iodine is titrated with a standard solution of sodium thiosulfate ($Na_2S_2O_3$).
    - **Indication:** Starch solution is used as the indicator.
    - **Why add starch late?** Starch is added only when the iodine solution has faded to a pale yellow colour (i.e., near the end-point). If added at the start when the iodine concentration is high, it forms a stable, insoluble blue-black complex that is slow to decolourise, making the end-point inaccurate.
    - **End-Point:** The colour change at the end-point is from **blue-black to colourless**.

4.5 Determining the K_a of a Weak Acid (CP 11)

This experiment determines the acid dissociation constant (K_a) for a weak acid by creating a pH titration curve.

- **Method:** A weak acid is titrated with a standard solution of a strong base (e.g., NaOH). The pH of the mixture is monitored throughout the titration using a pH meter.
- **The Half-Equivalence Point:** The most important point on the resulting graph is the half-equivalence point (the point where half the volume of base required for neutralisation has been added).
    - At this specific point, exactly half of the weak acid (HA) has been converted to its conjugate base (A^-). Therefore, **[HA] = [A^-]**.
    - In the K_a expression, $K_a = [H^+][A^-] / [HA]$, the $[A^-]$ and [HA] terms cancel out, leaving $K_a = [H^+]$.
    - Therefore, at the half-equivalence point, **pH = pK_a**. The pK_a can be read directly from the graph, and K_a can be calculated.
- **pH Meter Calibration:** Before use, a pH meter must be calibrated using standard buffer solutions of known pH to ensure its readings are accurate. Using a pH meter is more accurate and objective than using a chemical indicator, which relies on a subjective colour change.

These quantitative techniques are frequently combined with purification methods in the synthesis of new molecules.

5.0 Key Organic and Inorganic Preparations

Synthesis practicals are the culmination of your practical skills, requiring you to combine many of the techniques discussed previously—such as heating, separation, and purification—to create a target molecule. These preparations assess your understanding of the entire experimental process, from selecting reactants to obtaining a pure, dry product and assessing its final yield and purity.

5.1 Preparation of Aspirin (CP 16)

Aspirin (acetylsalicylic acid) is prepared by the esterification of the phenol group in 2-hydroxybenzoic acid.

- **Reaction:** 2-hydroxybenzoic acid is acetylated using ethanoic anhydride as the acetylating agent, with concentrated sulfuric or phosphoric acid as a catalyst.
- **Key Steps:**
    1. The reactants are gently warmed under **reflux** to ensure the reaction goes to completion without losing volatile substances.

2. The reaction mixture is then poured into ice water, causing the less soluble aspirin to precipitate out as a crude solid.
3. The crude product is collected by **suction filtration** using a Büchner funnel.
4. The aspirin is purified by **recrystallisation**, typically from a minimum volume of warm ethanol or an ethanol/water mixture.

- **Purity Assessment:** The purity of the final, dry product is determined by measuring its **melting point**. Pure aspirin has a sharp melting point of **135 °C**. An impure sample would melt at a lower temperature and over a broader range.

Note how this single preparation is a synoptic exercise, combining the core skills of **heating under reflux (2.1)**, **suction filtration (2.5)**, **recrystallisation (2.5)**, and purity assessment via **melting point analysis (2.6)**. Examiners expect you to see and explain these connections.

5.2 Preparation of a Transition Metal Complex (CP 14)

This practical involves the synthesis of the complex ion salt tetraamminecopper(II) sulfate.

- **Reaction:** Hydrated copper(II) sulfate reacts with an excess of concentrated ammonia in an aqueous solution. The ammonia molecules act as ligands, displacing the water ligands around the central copper(II) ion.
- **Key Steps:**
    1. Hydrated copper(II) sulfate is dissolved in a minimum amount of water.
    2. Concentrated ammonia is added (in a fume cupboard) until the initial precipitate redissolves, forming a deep blue solution of the complex.
    3. Ethanol is added to the solution, which reduces the solubility of the ionic complex salt, causing it to precipitate out as crystals, especially upon cooling in an ice bath.
    4. The crystals are collected by **suction filtration**, washed with a little cold ethanol, and dried.
- **Sources of Error in Yield:**
    - **Yield < 100%:** Product may be lost during transfers between vessels, some product may remain dissolved in the filtrate after crystallisation, or the reaction may not have gone to completion.

- **Apparent Yield > 100%:** This is almost always due to the final product not being completely dry. The mass of the residual solvent (water or ethanol) is included in the final mass of the crystals.

This preparation demonstrates how foundational skills are combined, requiring careful **precipitation control** by adding ethanol, efficient collection via **suction filtration (2.5)**, and an understanding of factors affecting **percentage yield**, such as incomplete crystallisation or a wet product.

With a firm grasp of these core techniques and their applications, we can now turn our attention to exam strategy and avoiding common errors.

6.0 Common Mistakes, Examiner Insights, and Exam Strategy

Success in the Unit 6 paper depends not only on knowing the procedures but also on avoiding common, costly errors and understanding precisely what examiners are looking for in your answers. This final section consolidates critical advice, highlighting frequent student mistakes and outlining a robust strategy to help you maximise your performance on exam day.

6.1 A Table of Frequent Student Errors and How to Avoid Them

The following table addresses common questions and misconceptions. Understanding the rationale behind each point is key to writing high-quality answers.

Common Mistake / Question	Correct Rationale / Procedure
Why is a *minimum volume* of hot solvent used in recrystallisation?	To create a saturated solution of the desired product. This ensures that upon cooling, the maximum amount of pure solid crystallises out, leading to a high yield. Using too much solvent would mean less product crystallises.
Why is the funnel pre-heated for hot filtration?	To prevent the dissolved solid from crystallising prematurely on the cold surface of the funnel or filter paper. This would lead to a significant loss of product and a lower final yield.
Why must the top of a reflux condenser be left open?	To prevent the build-up of pressure from the vaporising liquid. A sealed system would become pressurised upon heating and could explode, posing a serious safety risk.
Why are anti-bumping granules used?	To ensure smooth, even boiling. The granules provide a large surface area for bubbles to form on, preventing the

	formation of large bubbles and violent 'bumping' or 'flash boiling'.
Why is a water bath often used to heat flammable organic liquids?	To provide gentle, controllable heating and, most importantly, to prevent ignition. Using a direct Bunsen flame with a volatile, flammable substance is a major fire hazard.
When titrating iodine with thiosulfate, why is starch indicator added late?	Starch should only be added when the solution is pale yellow. If added at the start, when the iodine concentration is high, it forms a stable, insoluble starch-iodine complex that is very slow to break down, resulting in an inaccurate end-point.
Why is only dilute H_2SO_4 used in titrations with $KMnO_4$?	HCl cannot be used because MnO_4^- would oxidise the Cl^- ions to Cl_2 gas. Nitric acid cannot be used as it is also an oxidising agent and would react with the substance being analysed (e.g., Fe^{2+}). Sulfuric acid is stable and does not interfere with the redox reaction.

6.2 Exam Day Checklist and Strategy

Adopt a systematic approach when answering questions that require you to describe an experimental procedure.

- **Read the Question Carefully:** Before writing, identify the primary objective of the experiment. Are you making something, measuring a rate, finding a concentration, or identifying an unknown?
- **Name Specific Apparatus:** Use precise terminology. For accurate volume measurements, state "25.0 cm^3 volumetric pipette" and "burette," not just "pipette" or "measuring cylinder." For synthesis, mention a "pear-shaped flask" for reflux.
- **State All Measurements:** Be explicit about what you are measuring and with what instrument. For example: "Measure the mass of the solid using a 2 d.p. balance," or "Record the initial and final burette readings to 2 decimal places (±0.05 cm^3)."
- **Include Safety Precautions:** Always integrate relevant safety measures into your method. This is a guaranteed source of marks. Examples include: "Carry out the reaction in a fume cupboard as toxic gas is evolved," or "Wear gloves as concentrated acid is corrosive."
- **Control of Variables:** When describing an investigation into reaction rates, clearly state which variable you are changing (e.g., concentration of one reactant), which

variables must be kept constant (e.g., temperature, total volume, concentration of other reactants), and what you will be measuring (e.g., time taken for a colour change).

- **Be Precise with Observations:** Vague descriptions like "a precipitate formed" or "a colour change was seen" will not earn full marks. Be specific: "A dense white precipitate was formed," or "The solution changed colour from orange to green."
- **Justify Your Steps:** Where possible, briefly explain *why* a certain step is performed. This demonstrates a deeper understanding (e.g., "...add anhydrous sodium sulfate until the liquid turns clear to ensure it is dry").