

6 • Practical Skills in Chemistry II

Pearson Edexcel • International A-Level • Chemistry

Analysis, synthesis and the judgement between

WCH16 takes the bench work of Units 4 and 5 and asks you to defend it: which cation is this when two precipitates look alike, why the volume of one reagent need not be exact, which titres enter the mean, and what a straight line's gradient is worth in kJ mol^{-1} . The paper rewards the chemist who knows *why* each step happened – and it will happily test the scheme word-for-word.

WCH16/01 Practical Skills in Chemistry II is 1 hour 20 minutes, 50 marks, all compulsory, mostly on Units 4–5 but free to draw on Units 1–2. The assessed abilities match Unit 3's – practical problem-solving, methods, numeracy, apparatus – now applied to equilibria, kinetics, transition metals and organic synthesis.

The IA2 core practicals

Core practical	What it does	Key skill
9a Iodine–propanone rate	Quench samples in ice, titrate remaining iodine with thiosulfate	Quenching and titrimetric rate-following
9b Clock reaction	Harcourt–Esson: time to the starch endpoint; $1/t \propto$ initial rate	Timing, temperature control
10 Activation energy	Clock reaction across temperatures; plot $\ln(1/t)$ against $1/T$	Arrhenius graph: gradient = $-E_a/R$
11 K_a of a weak acid	pH measurements (or titration curve) of a weak acid	pH meter use, K_a from data
12 Electrochemical cells	Measure emf of metal/metal-ion half-cells joined by a salt bridge	Cell diagrams, conditions affecting emf
13a Redox titration: Fe^{2+}	Titrate iron(II) against potassium manganate(VII), acidified	Self-indicating endpoint (purple persists)
13b Redox titration: iodine	Iodine against sodium thiosulfate with starch	Accurate endpoint management
14 Transition metal complex	Prepare a complex salt from solution, crystallise, dry	Quantitative preparation and purity

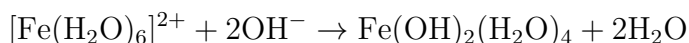
Core practical	What it does	Key skill
15 Inorganic/organic unknowns	The full A2 qualitative scheme	Complex-ion equations and deductions
16 Aspirin preparation	Salicylic acid + ethanoic anhydride; recrystallise; melting temperature	Yield, purification, purity checks

Cations at A2: precipitates, complexes, air

add aqueous NaOH dropwise, then in excess:

Cu^{2+}	pale blue ppt	in excess NH_3 : deep blue solution
Fe^{2+}	green ppt, browns in air	insoluble in excess
Fe^{3+}	rust-brown ppt	insoluble in excess
Mn^{2+}	pale brown ppt, darkens in air	insoluble in excess
NH_4^+	no ppt; NH_3 turns red litmus blue	on warming

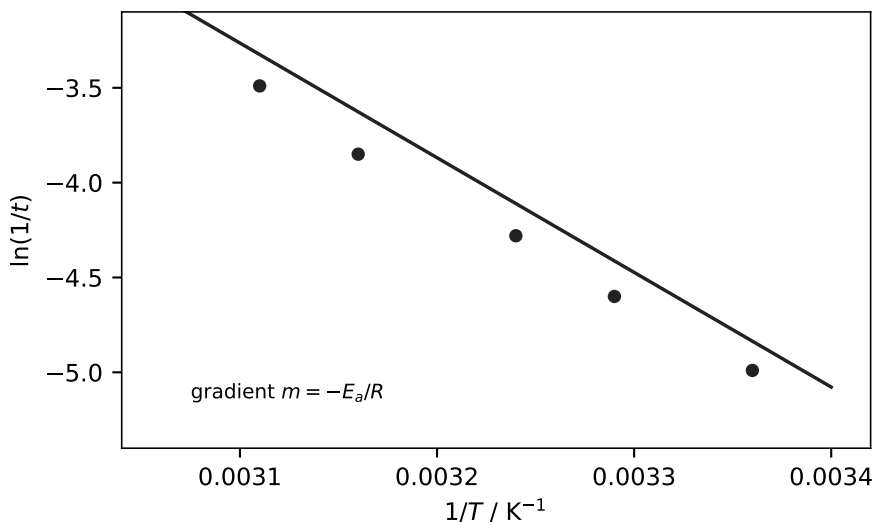
With aqueous sodium hydroxide: Cu^{2+} gives a pale blue precipitate (deep blue solution in excess ammonia); Fe^{2+} a **green** precipitate that browns in air; Fe^{3+} a rust-brown precipitate; Mn^{2+} a **pale brown precipitate that darkens on standing** – the 2025 paper's cation; NH_4^+ no precipitate, but warming releases ammonia that turns damp red litmus blue. Ionic equations may use either form:



Dissolving iron(III) hydroxide in sulfuric acid gives the yellow-brown $[\text{Fe}(\text{H}_2\text{O})_6]^{3+}$ complex. An unknown with M_r 197.9 and a $\text{Mn}^{2+}/\text{Cl}^-$ analysis is $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$: $197.9 - (54.9 + 71.0) = 72.0 = 4 \times 18.0$. A salt holding two cations (green ppt + ammonia on warming + white ppt with acidified barium chloride) is Mohr's salt, $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2$.

The clock reaction and activation energy

Thiosulfate is added in a **small known amount** to the peroxodisulfate–iodide mixture: it reacts with the iodine as it forms ($2\text{S}_2\text{O}_3^{2-} + \text{I}_2 \rightarrow \text{S}_4\text{O}_6^{2-} + 2\text{I}^-$), so the **colour change is delayed** until the thiosulfate is used up (a **clock reaction**) – then the next iodine turns the starch **blue-black** (from colourless). Because the same small amount of iodine is waiting at every endpoint, $1/t$ is proportional to the initial rate. Two technique marks hide in the 2025 scheme: a **burette** (not a measuring cylinder) adds even the starch, because keeping the **total volume identical keeps the reactant concentrations constant** between runs – not because burettes are more precise; and both tubes equilibrate in the water bath before mixing, with the temperature recorded *in the reaction mixture*.



Completing the table is arithmetic discipline: $T(\text{K}) = T(^{\circ}\text{C}) + 273$, $1/t$ to 3 significant figures, $\ln(1/t)$ to 2 decimal places. The straight line of $\ln(1/t)$ against $1/T$ has gradient $m = -E_a/R$ with E_a the **activation energy** :

$$E_a = -mR = 6045 \times 8.31 = 50\,234 \text{ J mol}^{-1} = 50.2 \text{ kJ mol}^{-1}$$

Positive value, kJ mol^{-1} , no negative sign – the sign is already in the gradient.

Titration that build an answer

The calcium hydroxide experiment shows the full chain. Why stand for a day? To reach **equilibrium** – a **saturated solution**. Indicator: **methyl orange** (yellow $\rightarrow$ orange) or **phenolphthalein** (pink $\rightarrow$ colourless). Titres 9.95, 9.55 and 9.45 cm^3 : the first is **not concordant** and is excluded – mean $= (9.55 + 9.45)/2 = 9.50 \text{ cm}^3$. The expected titre from the data book (1.56 g dm^{-3}):

$$c = 1.56/74.1 = 0.0211 \text{ mol dm}^{-3}; \quad n = 0.0211 \times 0.0250 = 5.26 \times 10^{-4} \text{ mol}$$

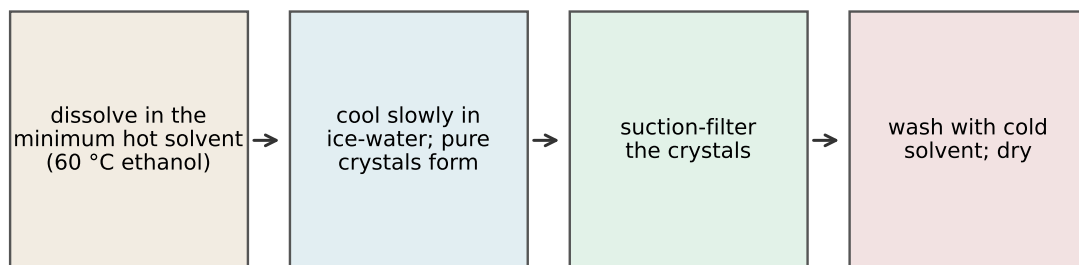
$$n(\text{HCl}) = 2 \times 5.26 \times 10^{-4} = 1.05 \times 10^{-3} \text{ mol}; \quad V = 10.55 \text{ cm}^3$$

A mean titre *lower* than expected, given that solubility falls as temperature rises, means the experiment ran **warmer than 20 °C**. Finally $K_c = [\text{Ca}^{2+}][\text{OH}^-]^2 = 0.0190 \times (0.0380)^2 = 2.74 \times 10^{-5} \text{ mol}^3 \text{ dm}^{-9}$ – units derived, not remembered.

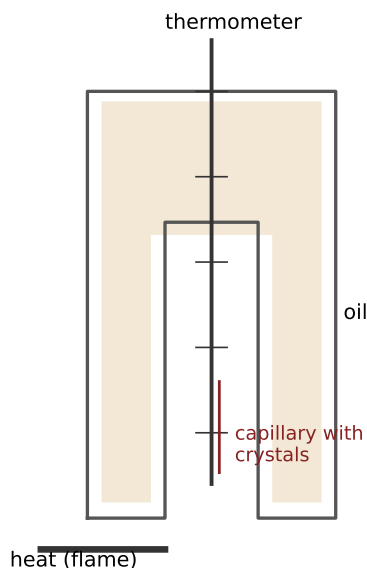
Preparing and purifying an organic solid

The phenyl benzoate preparation (benzoyl chloride + phenol in sodium hydroxide) is the template.

- **Safety:** gloves for weighing phenol (toxic by skin absorption); **fume cupboard** because **benzoyl chloride is toxic by inhalation**.
- **Why a volume may be inexact:** show by calculation which reagent limits. $9 \text{ cm}^3 \times 1.21 = 10.89 \text{ g} = 0.0775 \text{ mol}$ benzoyl chloride, against $5.00/94.0 = 0.0532 \text{ mol}$ phenol – benzoyl chloride is in **excess**, so its exact volume does not affect the yield.
- **Washing with water** removes the **soluble** impurities: excess benzoyl chloride (hydrolysed), HCl, NaOH, NaCl. Never say “insoluble impurities” – the scheme rejects it.
- **Recrystallisation** : dissolve in the minimum hot solvent – at 60 °C, below ethanol’s boiling point and, crucially, below the product’s melting temperature (71 °C), **so the crystals do not melt**; cool in ice-water; filter; wash with **cold** solvent, which removes soluble impurities while the crystals barely dissolve. A sharp **melting temperature** at the data-book value certifies purity.



impurities stay dissolved at every stage; the product is only soluble hot



Worked check (yield). $n(\text{phenol}) = 5.00/94.0 = 0.0532 \text{ mol}$; 1:1 stoichiometry; theoretical mass = $0.0532 \times 198.0 = 10.5 \text{ g}$; at 65.0 % yield, mass = 6.85 g.

What written practice cannot do

As throughout this qualification, the written paper samples your *understanding* of practical work: the observations, the reasons, the arithmetic and the judgement. The manipulative craft – a clean titration endpoint, a working Thiele tube, a quantitative transfer – is built in class through the core practicals themselves.

Check yourself

1. A pale brown precipitate that darkens on standing in air: which cation, and what darkens it?
2. Write two acceptable ionic equations for the green precipitate when NaOH(aq) meets an Fe^{2+} solution.
3. Salt B gives a green precipitate with NaOH , ammonia on warming, and a white precipitate with acidified barium chloride. Deduce the salt.
4. Why is thiosulfate added to a clock reaction, and what ends the timing?
5. Titres 9.95, 9.55, 9.45 cm^3 – which enter the mean, and what is it?
6. From a $\ln(1/t) - 1/T$ gradient of -6045 K , calculate E_a with unit.
7. Why dissolve the crude solid at 60°C rather than at ethanol's boiling point?
8. Give two things a water wash removes from crude phenyl benzoate, and one phrase the scheme rejects.
9. Sketch or describe the melting-temperature apparatus, naming three labelled parts.

10. 5.00 g phenol (Mr 94.0) at 65.0 % yield gives what mass of phenyl benzoate (Mr 198.0)?

Answers: 1 Mn^{2+} ; air oxidises the pale brown $\text{Mn}(\text{OH})_2$ to a darker manganese(III/IV) oxide/hydroxide; 2 $\text{Fe}^{2+} + 2\text{OH}^- \rightarrow \text{Fe}(\text{OH})_2$, or $[\text{Fe}(\text{H}_2\text{O})_6]^{2+} + 2\text{OH}^- \rightarrow \text{Fe}(\text{OH})_2(\text{H}_2\text{O})_4 + 2\text{H}_2\text{O}$; 3 $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 - \text{NH}_4^+$, Fe^{2+} and SO_4^{2-} ; 4 it reacts with iodine as it forms, delaying the starch colour until a fixed small amount of iodine has been produced; the blue-black colour; 5 9.55 and 9.45 (concordant within 0.2 cm^3); mean 9.50 cm^3 ; 6 $E_a = 6045 \times 8.31 = 50.2 \text{ kJ mol}^{-1}$; 7 ethanol's boiling point (78°C) is above the product's melting temperature (71°C) – at the boil the crystals would melt; 60°C dissolves without melting; 8 removes excess benzoyl chloride, HCl , NaOH or NaCl (soluble) – "insoluble impurities" is rejected; 9 Thiele tube (or boiling tube/beaker) filled with oil, labelled thermometer in the liquid, capillary tube of crystals attached, heated with a flame (not electrical); 10 $n = 5.00/94.0 = 0.0532 \text{ mol}$; $0.0532 \times 198.0 \times 0.650 = 6.85 \text{ g}$.