Unit 6: Practical Skills in Chemistry II

Pearson WCH16 · core practicals 9–16 (Units 4–5) · written practical skills; bench manipulation stays class work.

Defend every step

Your turn

Two precipitates look alike, one volume is only approximate, and a graph gives a gradient.

A. How do you tell the two cations apart?

B. Why does one reagent's volume not need to be exact?

C. What is the gradient worth in kJ mol^{-1}?

The A2 core practicals

Core practical	**Key skill**
9 iodine–propanone rate; **clock reaction**	quenching and titrating; $1/\mathrm{t} \propto$ initial rate
10 **activation energy**	Arrhenius plot: **gradient** $= -E_a/R$
11 Ka of a weak acid	pH meter readings to Ka
12 electrochemical cells	half-cells, salt bridge, emf
13 redox titrations	self-indicating manganate(VII); iodine with starch
14 transition metal complex	prepare, crystallise and dry a complex salt
15 and 16 unknowns; aspirin	qualitative scheme; yield, recrystallisation, melting point

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Cations with sodium hydroxide

Cation	**With NaOH(aq)**	**Then**
Cu^{2+}	pale blue **precipitate**	deep blue solution in **excess** ammonia
Fe^{2+}	green precipitate	turns brown in air
Fe^{3+}	rust-brown precipitate	no change
Mn^{2+}	pale brown precipitate	darkens on standing in air
$NH_4{}^+$	no precipitate	warm; ammonia turns damp red **litmus** blue

Equations and deductions

1. **Simple** $\mathrm{Fe^{2+} + 2OH^- \rightarrow Fe(OH)_2}$
2. **Complex** $\mathrm{[Fe(H_2O)_6]^{2+} + 2OH^- \rightarrow Fe(OH)_2(H_2O)_4 + 2H_2O}$
3. **Water** Mn^{2+} and Cl^-, Mr 197.9: $197.9 - (54.9 + 71.0) = 72.0 = 4 \times 18.0$, so $MnCl_2 \cdot 4H_2O$.
4. **Two cations** Green **precipitate**, ammonia on warming, white $BaSO_4$ means $(NH_4)_2Fe(SO_4)_2$.

Decision

Every observation must be used in the deduction.

How the clock reaction works

Peroxodisulfate oxidises iodide to iodine.

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A little thiosulfate turns it straight back.

↓

Thiosulfate used up: **starch** turns blue-black.

↓

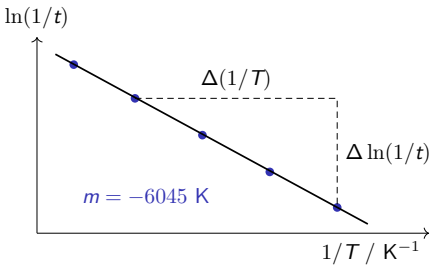
Same iodine each run, so $1/t \propto$ initial rate.

A **burette** for every volume keeps the total volume, so the concentrations, the same.

Activation energy from a gradient

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

- Plot ln(1/t) against 1/T, with T in kelvin.
- **Gradient** = −Ea/R.



Saturated calcium hydroxide, titrated

1. **Titres** 9.95, 9.55, 9.45 cm^3; the 9.95 is not **concordant**.
2. **Mean** $(9.55 + 9.45)/2 = 9.50$ cm^3
3. **Expected** $1.56/74.1 = 0.0211$ mol dm^{-3}; $\times 0.0250$ $dm^3 = 5.26 \times 10^{-4}$ mol; $\times 2$ HCl $\div$ $0.100 = 10.5$ cm^3
4. **Judge** Lower than expected; **solubility** falls as temperature rises, so it was warmer than 20 °C.

Decision

$K_c = 0.0190 \times 0.0380^2 = 2.74 \times 10^{-5}$ mol^3 dm^{-9}; derive the units.

Preparing phenyl benzoate

1. **Safety** Gloves for phenol (absorbed through skin); **fume cupboard** for toxic benzoyl chloride.
2. **Excess** 9 $cm^3 \times 1.21 = 10.89$ g $= 0.0775$ mol benzoyl chloride; phenol $5.00/94.0 = 0.0532$ mol.
3. **Wash** Water removes **soluble** impurities — **excess** benzoyl chloride, HCl, NaOH, NaCl.
4. **Yield** $0.0532 \times 198.0 = 10.5$ g theoretical; at 65.0 %, 6.85 g.

Decision

Never write "insoluble impurities": the scheme rejects it.

Recrystallise, then check the melting point

Dissolve in the minimum hot ethanol at 60 °C, below the product's 71 °C, so it does not melt.

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Cool in ice-water so crystals form.

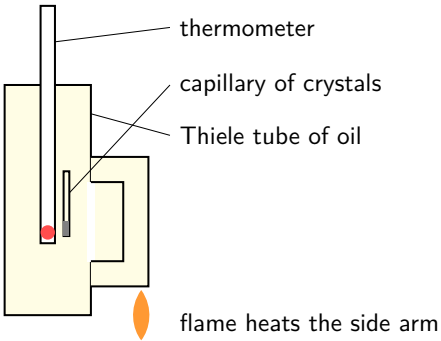
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Suction-filter and wash with cold **solvent**, which removes **soluble** impurities.

↓

Dry, then measure the **melting temperature** in a Thiele tube.

The melting-temperature apparatus



A pure solid melts sharply at its data-book value; impurities lower it and widen the range.

- Name the labelled parts.
- Heat slowly near the expected value.

Written practice has limits

A clean titration end point, a working **Thiele tube** and a quantitative transfer are learned at the bench.

- The paper samples understanding.
- The craft is built in class.

Check yourself

Your turn

Answer without notes, then check.

A. A pale brown precipitate darkens on standing. Which cation, and why?

B. Why is thiosulfate added to a clock reaction, and what ends the timing?

C. From a gradient of -6045 K, calculate Ea with its unit.

D. Why dissolve the crude solid at 60 °C rather than at ethanol's boiling point?

E. 5.00 g phenol at 65.0 % yield gives what mass of phenyl benzoate?