Answers to Practicals

Section 6

The practical part of your chemistry course is an important as the theory. Your examiners expect you to have done a comprehensive programme of practical work and will test you on your knowledge of practical chemistry.

Although you may be working in pairs for some of these investigations, it is important that you make your own observations, record all the results and process the data yourself. Sample results and model answers to these investigations are on the CD.

Contents

Safety notes

Those substances identified as CORROSIVE will quickly make holes in your skin or clothes. If you get even a single drop on your skin, wash it off immediately (within a few seconds). Those susbtances identified as TOXIC are particularly poisonous. You should wash your hands after handling them. Most dilute solutions are safe under normal circumstances, but can still harm you if you get some in your eye or mouth.

Safety glasses should be worn for all practical work
Always fill pipettes with a pipette filler.

Inv 1.1 Redox reactions: some examples

Many redox reactions involve colour changes. The reactions below illustrate the behaviour of some common redox reagents.

Carry out each reaction using CLEAN test tubes and SMALL quantities.

Reaction one (iron powder and copper sulfate solution)

1 a To 2 mL of copper sulfate solution add half a spatula full of iron powder, mix well and allow to stand.

Observations: Solution changed from blue to colourless and a red/brown ppt forms.

b Use a dropper to remove most of the liquid and add it to 2 mL of dilute sodium hydroxide solution.

Observation and conclusion: A dark green precipitate forms so Fe^{2+} present.

c Add two drops of concentrated nitric acid (corrosive) to the solid remaining in the test tube.

Observation and conclusion: A brown gas is given off and the liquid turns blue. The solid is copper metal.

d What species was oxidised in this reaction? Fe To what? Fe^{2+}

e Write the oxidation half-equation.

$Fe(s) \rightarrow Fe^{2+}(aq) + 2e^-$

f What species was reduced in this reaction? Cu^{2+} To what? Cu.

g Write the reduction half-equation.

$Cu^{2+}(aq) + 2e^- \rightarrow Cu(s)$

h Combine the two half-equations into an ionic equation.

$Fe(s) + Cu^{2+}(aq) \rightarrow Fe^{2+}(aq) + Cu(s)$

Reaction two (potassium dichromate and ethanol)

2 a Place 2 mL of potassium dichromate solution in a test tube, add 2 mL of dilute sulfuric acid and warm in a water bath for two minutes. Then add 1 mL of ethanol.

Observations: The orange solution slowly turns green.

b Chromium ions you may have met are: CrO_4^{2-} (yellow), $Cr_2O_7^{2-}$ (orange), Cr^{3+} (green), Cr^{2+} (blue). Identify the reacting and product chromium ions for the reaction in a.

Reactant species: $Cr_2O_7^{2-}$ Product species: Cr^{3+}

c Write the ion-electron half-equation for the chromium species and state whether chromium has been oxidised or reduced.

$Cr_2O_7^{2-} + 14H^+ + 6e^- \rightarrow 2Cr^{3+} + 7H_2O$ reduced

d Assume that the ethanol (CH_3CH_2OH) reacts to form ethanal (CH_3CHO). Write the ion-electron half-equation for the conversion of ethanol to ethanal and state whether this is oxidation or reduction.

$CH_3CH_2OH \rightarrow CH_3CHO + 2H^+ + 2e^-$ oxidation

e Combine these two half-equations to form an ionic redox equation.

$Cr_2O_7^{2-}(aq) + 3CH_3CH_2OH(aq) + 8H^+(aq) \rightarrow 2Cr^{3+}(aq) + 3CH_3CHO(aq) + 7H_2O(l)$

Reaction three (acidified hydrogen peroxide and potassium permanganate) PowerPoint

3 a To 2 mL of 6% hydrogen peroxide add 2 mL of dilute sulfuric acid followed by a few drops of potassium permanganate solution.

Observations: The purple solution turns colourless. (Bubbles of colourless gas formed)

b Manganese species you have met include: Mn^{2+} (colourless/very pale pink), Mn^{3+} (red), MnO_2 (brown), MnO_4^{2-} (green), MnO_4^- (purple). Identify the reactant and product manganese species.

Reactant species: MnO_4^- (purple) Product species: Mn^{2+} (colourless)

c Write the ion-electron half-equation for the manganese species and state whether it is oxidation or reduction.

$MnO_4^-(aq) + 8H^+(aq) + 5e^- \rightarrow Mn^{2+}(aq) + 4H_2O(l)$ reduction

d Hydrogen peroxide can be oxidised to form oxygen gas or reduced to form water. Decide whether it has been oxidised or reduced and write the appropriate ion-electron half equation.

Oxidised: $H_2O_2(aq) \rightarrow O_2(g) + 2H^+(aq) + 2e^-$

e Combine these two half-equations to form an ionic redox equation.

$2MnO_4^-(aq) + 5H_2O_2(aq) + 6H^+ \rightarrow 2Mn^{2+}(aq) + 5O_2(g) + 8H_2O(l)$

Reaction four (iron(II) sulfate and neutral potassium permanganate)

4 a To 2 mL of iron(II) sulfate solution add a few drops of potassium permanganate solution.

Observations: The purple colour disappears and a brown precipitate forms

b Add a drop of potassium thiocyanate solution.

Observation and conclusion: Solution turns blood-red so Fe^{3+} is present.

c Refer to **3b** to identify the reactant and product manganese species.

Reactant species: MnO_4^- (purple) Product species: Mn^{2+} (colourless)

d Write the ion-electron half-equation for the manganese species and state whether it is oxidation or reduction.

$MnO_4^-(aq) + 4H^+(aq) + 3e^- \rightarrow MnO_2(s) + 2H_2O(l)$ reduced

e Write the relevant ion-electron half equation for the Fe^{2+} reaction.

$Fe^{2+}(aq) \rightarrow Fe^{3+}(aq) + e^-$

f Combine these two half-equations to form an ionic redox equation.

$MnO_4^-(aq) + 4H^+(aq) + 3Fe^{2+}(aq) \rightarrow MnO_2(s) + 2H_2O(l) + 3Fe^{3+}(aq)$

Reaction five (oxalic acid and potassium permanganate)

5 **a** To 2 mL of oxalic acid solution (toxic) add 2 mL of dilute sulfuric acid solution and warm in a water bath for 2 minutes, then add a few drops of potassium permanganate solution.

Observations: The solution changes from purple to colourless + a colourless gas produced.

b Use a dropper to take a sample of gas from just above the surface of the liquid. Bubble this gas through 2 mL of limewater.

Observation and conclusion: Limewater turns cloudy so gas is CO_2.

c Refer to **3b** to identify the reactant and product manganese species.

Reactant species: MnO_4^- (purple) Product species: Mn^{2+} (colourless)

d Write the ion-electron half-equation for the manganese species and state whether it is oxidation or reduction.

$MnO_4^-(aq) + 8H^+(aq) + 5e^- \longrightarrow Mn^{2+}(aq) + 4H_2O(l)$ reduction

e Oxalic acid is present as the $C_2O_4^{2-}$ ion. What does this ion form in this reaction? CO_2

f Write the ion-electron half-equation for the $C_2O_4^{2-}$ reaction and state whether it is oxidation or reduction.

$C_2O_4^{2-}(aq) \longrightarrow 2CO_2(g) + 2e^-$ oxidation

g Combine these two half-equations to form an ionic redox equation.

$2MnO_4^-(aq) + 5C_2O_4^{2-}(aq) + 16H^+(aq) \longrightarrow 2Mn^{2+}(aq) + 10CO_2(g) + 8H_2O(l)$

Reaction six (alkaline potassium permanganate) PowerPoint

6 **a** To 2 mL of sodium hydrogen sulfite solution add 4 drops of potassium permanganate solution followed by 4 mL of dilute sodium hydroxide solution.

Observations: The solution goes from purple to green.

b In a separate test tube place 2 mL of dilute hydrochloric acid and 1 mL of barium chloride solution. Add a few drops of your solution from a.

Observation and conclusion: A white precipitate forms so SO_4^{2-} present.

c Write the oxidation ion-electron half equation for *alkaline conditions*.

$HSO_3^-(aq) + 3OH^-(aq) \longrightarrow SO_4^{2-}(aq) + 2H_2O(l) + 2e^-$

d Refer to **3b** to identify the reactant and product manganese species.

Reactant species: MnO_4^- (purple) Product species: MnO_4^{2-} (green)

e Write the reduction ion-electron half equation in *alkaline conditions*.

$MnO_4^-(aq) + e^- \longrightarrow MnO_4^{2-}(aq)$

f Combine these two half-equations to form an ionic redox equation.

$HSO_3^-(aq) + 3OH^-(aq) + 2MnO_4^- \longrightarrow SO_4^{2-}(aq) + 2H_2O(l) + 2MnO_4^{2-}(aq)$

Inv 1.2 Redox reactions involving the halogens

Group 17 elements are important redox reagents which exhibit a range of oxidation states.

Carry out each reaction using CLEAN test tubes and SMALL quantities.
Note ALL observations. Identify new substances if you can.

Reaction one (lead(IV) oxide and concentrated hydrochloric acid) PowerPoint

1 **a** To 1 mL of concentrated hydrochloric acid (corrosive) in a test tube add a rice-grain sized sample of solid lead(IV) oxide (toxic) powder. Hold a piece of damp starch-iodide paper near the top of the test tube.

Observation and conclusion: A gas is given off that turns starch-iodide paper blue-black then bleaches it white. It is Cl_2.

b Use a dropper to remove a few drops of the solution from a then add to them 1 mL of dilute potassium iodide solution

Observation and conclusion: A bright yellow ppt forms. Pb^{2+} is present.

c Write two ion-electron half equations and hence a full ionic reaction for the reaction in **a**.

$2Cl^-(aq) \rightarrow Cl_2(g) + 2e^-$

$PbO_2(s) + 4H^+(aq) + 2e^- \rightarrow Pb^{2+}(aq) + 2H_2O(l)$

$PbO_2(s) + 2Cl^-(aq) + 4H^+(aq) \rightarrow Pb^{2+}(aq) + Cl_2(g) + 2H_2O(l)$

Reaction two (potassium iodide and hydrogen peroxide)

2 **a** To 2 mL of dilute potassium iodide solution add 2 mL of dilute sulfuric acid solution followed by 1 mL of 6% hydrogen peroxide.

Observations: The colourless solution turns a yellow-brown colour.

b Add a few drops of starch solution.

Observation and conclusion: A blue-black colour indicates I_2 is present.

c What species was oxidised during the above redox reaction? $I^-(aq)$

d Write the oxidation half equation.

$2I^-(aq) \rightarrow I_2(aq) + 2e^-$ $\quad (I_2(aq) + I^-(aq) \rightarrow I_3^-(aq))$

e What was the oxidising agent during the above redox reaction? H_2O_2

f Write the reduction half equation.

$H_2O_2(aq) + 2H^+(aq) + 2e^- \rightarrow 2H_2O(l)$

g Combine these two half-equations to form an ionic redox equation.

$2I^-(aq) + H_2O_2(aq) + 2H^+(aq) \rightarrow I_2(aq) + 2H_2O(l)$

Reaction three (sodium thiosulfate and iodine) PowerPoint

3 **a** To 2 mL of sodium thiosulfate add 1 mL of iodine solution.

Observations: The yellow-brown solution turns colourless.

b Add a few drops of silver nitrate solution.

Observation and conclusion: A pale yellow precipitate forms so I^- is present.

c Write the reduction half-equation.

$I_2(aq) + 2e^- \rightarrow 2I^-(aq)$

d $S_2O_3^{2-}$ is oxidised to the tetrathionate ion, $S_4O_6^{2-}$. What colour is this ion? colourless

e Write the oxidation half equation.

$2S_2O_3^{2-}(aq) \rightarrow S_4O_6^{2-}(aq) + 2e^-$

f Combine the two half-equations into a full ionic equation.

$2S_2O_3^{2-}(aq) + I_2(aq) \rightarrow S_4O_6^{2-}(aq) + 2I^-(aq)$

Reaction four (potassium iodate and sodium sulfite)

4 **a** Acidify 2 mL of potassium iodate solution with 2 mL of dilute hydrochloric acid solution, then add 3 mL of sodium sulfite solution.

Observations: The colourless soln turns dark brown.

b Withdraw 1 mL of solution from a and add it to 1 mL of barium chloride solution.

Observation and conclusion: A white ppt forms so SO_4^{2-} present.

c Add 1 drop of starch to the remaining solution from **a**.

Observation and conclusion: A blue-black colour indicates that I_2 present

d Write the iodate half-equation and state whether the iodate ion was oxidised or reduced.

$2IO_3^-(aq) + 12H^+(aq) + 10e^- \rightarrow I_2(aq) + 6H_2O(l)$ reduced

e Write the sulfite half-equation and state whether the sulfite ion was oxidised or reduced.

$SO_3^{2-}(aq) + H_2O(l) \rightarrow SO_4^{2-}(aq) + 2H^+(aq) + 2e^-$ oxidised

f Combine the two half-equations into a full ionic equation.

$2IO_3^-(aq) + 5SO_3^{2-}(aq) + 2H^+(aq) \rightarrow I_2(aq) + 5SO_4^{2-}(aq) + H_2O(l)$

Note: If your soln went colourless in (a), then the main reduction product is $I^-(aq)$.
Even a trace of I_2 will go blue-black in (c), but your eqn for (d) should be

$IO_3^- + 6H^+ + 6e^- \rightarrow I^- + 3H_2O$

and (f) $IO_3^- + 3SO_3^{2-} \rightarrow I^- + 3SO_4^{2-}$

Reaction five (potassium iodide and potassium iodate)

5 a To 4 mL of potassium iodide solution add 2 mL of dilute sulfuric acid solution followed by 2 mL of potassium iodide solution.

Observations: The colourless solution turns brown.

b Add a drop of starch solution.

Observation and conclusion: A blue-black colour appears so I_2 present.

c What is formed when iodide ions are oxidised? I_2

d What is formed when iodate ions are reduced? I_2 (and then I^-)

e Write two ion-electron half equations and hence a full ionic reaction for the reaction in **a**.

$2I^-(aq) \rightarrow I_2(aq) + 2e^-$

$2IO_3^-(aq) + 12H^+(aq) + 10e^- \rightarrow I_2(aq) + 6H_2O(l)$

$5I^-(aq) + IO_3^-(aq) + 6H^+ \rightarrow 3I_2(aq) + 3H_2O$

Reaction six (potassium iodide and copper sulfate) PowerPoint

6 a To 2 mL of potassium iodide solution add 1 mL of copper sulfate solution. Filter the resulting mixture.

Observations: The blue colour disappears and a cloudy/milky brown colour forms. The filtrate is orange/brown and a white solid appears on the brown-stained filter paper.

b Copper species you may have met include blue $Cu^{2+}(aq)$, black CuO(s), red $Cu_2O(s)$, white CuI(s) and brown/pink Cu(s). Identify the reactant and product copper species in **a**.

Reactant: Cu^{2+} blue Product: CuI(s) white

c Write an ion-electron half equation for the copper reaction and state whether it is oxidation or reduction.

$I^-(aq) + Cu^{2+}(aq) + e^- \rightarrow CuI(s)$ reduction

d What is the product of the iodide reaction? I_2

e Write an ion-electron half equation for the iodide reaction.

$2I^-(aq) \rightarrow I_2(aq) + 2e^-$

Combine the two half equations into one full ionic equation.

$2Cu^{2+}(aq) + 4I^-(aq) \rightarrow 2CuI(s) + I_2(aq)$

(also $I_2(aq) + I^-(aq) \rightarrow I_3^-(aq)$)

Inv 2.1 Preparation of standard iron(II) ammonium sulfate

Iron(II) ammonium sulfate is suitable as a primary standard for quantitative analysis because it is a stable solid easily obtained in a pure form. It reacts with a number of oxidising agents. The solution prepared here can be used to standardise potassium permanganate solution (Inv 2.2).

1 Calculate the molar mass of $FeSO_4(NH_4)_2SO_4.6H_2O$. $M(FeSO_4(NH_4)_2SO_4.6H_2O)$ = 392 g mol^{-1}

2 What size of volumetric flask will you use? $V(FeSO_4(NH_4)_2SO_4.6H_2O)$ = 200.0 mL

3 What is the required concentration of the iron(II) ammonium sulfate solution? $c(FeSO_4(NH_4)_2SO_4.6H_2O)$ = 0.100 mol L^{-1}

4 Write the formula relating the amount in moles, concentration and volume. $n = cV$

5 Calculate the amount, in moles, of iron(II) ammonium sulfate solution required. $n(FeSO_4(NH_4)_2SO_4.6H_2O)$ = 0.0200 mol

$n = cV$
$= 0.100 \text{ mol L}^{-1} \times 200.0 \times 10^{-3} \text{ L}$
$= 0.0200 \text{ mol}$

6 Write the formula relating the amount in moles, mass and molar mass. $n = \frac{m}{M}$ $m = nM$

7 Calculate the mass of iron(II) ammonium sulfate required. $m(FeSO_4(NH_4)_2SO_4.6H_2O)$ = 7.84 g

$m = nM$
$= 0.0200 \text{ mol} \times 392 \text{ g mol}^{-1}$
$= 7.84 \text{ g}$

8 Weigh a small, clean, dry beaker. mass = —

9 Add about (within about 0.3 g) the required mass of iron(II) ammonium sulfate required and reweigh. mass = —

10 Calculate the mass of iron(II) ammonium sulfate used. $m(FeSO_4(NH_4)_2SO_4.6H_2O)$ = 7.758 g

11 Calculate the number of moles of iron(II) ammonium sulfate used. $n = \frac{m}{M}$ $n(FeSO_4(NH_4)_2SO_4.6H_2O)$ = 0.0189 mol

$n = \frac{7.758 \text{ g}}{392 \text{ g mol}^{-1}}$
$= 0.0189 \text{ mol}$

12 Dissolve the powder in about half a beaker of distilled water. Rinse the clean volumetric flask with distilled water. Use a funnel to carefully transfer the iron(II) ammonium sulfate solution to the volumetric flask and rinse in any remainder using distilled water. Make sure to wash down the sides of the funnel. Shake until dissolved and make up to the mark. Stopper and shake to mix thoroughly.

13 Calculate the exact concentration of the solution. $c(FeSO_4(NH_4)_2SO_4.6H_2O)$ = 0.0989 mol L^{-1}

$c = \frac{n}{V}$
$= \frac{0.0189 \text{ mol}}{200.0 \times 10^{-3} \text{ L}}$
$= 0.0989 \text{ mol L}^{-1}$

Inv 2.2 Standardising potassium permanganate solution PowerPoint

We can use a standard solution of iron(II) ammonium sulfate to find the concentration of an unknown solution of potassium permanganate.

1 Rinse and fill a burette with the potassium permanganate solution (approximately 0.02 mol L^{-1}).

2 Using a pipette filler, pipette out 20.0 mL samples of the iron(II) ammonium sulfate solution into conical flasks and add 20 mL of dilute sulfuric acid to each flask.

$V(Fe^{2+})$ = 20.0 mL

3 What is the concentration of this iron(II) ammonium sulfate solution?

$c(Fe^{2+})$ = 0.0989 mol L^{-1}

4 Calculate the amount, in moles, of iron(II) ammonium sulfate solution used in each titre.

$n(Fe^{2+})$ = 1.978×10^{-3} mol

$n = cV$
$= 0.0989 \text{ mol } L^{-1} \times 20.0 \times 10^{-3} \text{ L}$
$= 1.978 \times 10^{-3} \text{ mol}$

5 Titrate the permanganate solution into the acidified iron(II) ammonium sulfate solution until the pink colour remains. Repeat until 3 concordant results are obtained.

Burette reading	1st titration	2nd titration	3rd titration	4th titration	5th titration
Initial reading mL	3.8	23.7	19.1	23.8	19.4
Final reading mL	23.6	43.9	38.8	43.5	37.2
Titre mL	19.8	~~20.2~~	19.7	19.7	19.8

6 Calculate the average of the concordant titres.

$V(MnO_4^-)$ = 19.75 mL

$\frac{19.8 + 19.7 + 19.7 + 19.8}{4} = 19.75$

The ionic equation for this redox reaction is

$$5Fe^{2+}(aq) + MnO_4^-(aq) + 8H^+(aq) \longrightarrow 5Fe^{3+}(aq) + Mn^{2+}(aq) + 4H_2O(l)$$

7 What is the ratio of MnO_4^- to Fe^{2+}?

$\frac{n(MnO_4^-)}{n(Fe^{2+})} = \frac{1}{5}$

$n(MnO_4^-) = \frac{n\,Fe^{2+}}{5}$

$n(MnO_4^-)$ = $\frac{1}{5}$ $n(Fe^{2+})$

8 Calculate the amount, in moles, of permanganate reacting.

$n(MnO_4^-)$ = 3.956×10^{-4} mol

$n(MnO_4^-) = \frac{1}{5} \times n(Fe^{2+})$
$= \frac{1.978 \times 10^{-3} \text{ mol}}{5}$
$= 3.956 \times 10^{-4} \text{ mol}$

9 Calculate the concentration of the permanganate solution.

$c(MnO_4^-)$ = 0.0200 mol L^{-1}

$c(MnO_4^-) = \frac{n}{V}$
$= \frac{3.956 \times 10^{-4} \text{ mol}}{19.75 \times 10^{-3} \text{ L}}$
$= 0.0200 \text{ mol } L^{-1}$

Inv 2.3 Standardising potassium permanganate solution with oxalic acid PowerPoint

Oxalic acid, used to reduce potassium permanganate, is poisonous. Pipette fillers must be used.

1 Weigh accurately about 1.3 g of oxalic acid (toxic) into a small,

m(oxalic) = 1.307 g

2 Dissolve the oxalic acid in distilled water and transfer the solution to a 200.0 mL volumetric flask. Rinse the beaker into the flask several times. Mix well, make up to the mark with distilled water and mix again. Label your flask.

$V(Fe^{2+})$ = 200.0×10^{-3} L

3 Calculate the exact concentration of this solution, M(oxalic acid) = 126 g mol^{-1}.

c(oxalic) = 0.05187 mol L^{-1}

$c(\text{oxalic}) = \frac{m}{MV} = \frac{1.307\text{ g}}{126\text{ g mol}^{-1} \times 200.0 \times 10^{-3}\text{ L}}$

$= 0.05187\text{ mol L}^{-1}$

4 Rinse and fill a burette with the permanganate solution (approximately 0.02 mol L^{-1}).

5 Using a pipette filler, pipette out 20.0 mL (or 25.0 mL) samples of the oxalic acid solution into conical flasks and add 20 mL of dilute sulfuric acid to each flask. Heat each solution on a water bath or over a Bunsen flame until it is about 80 °C.

V(oxalic) = 20.0×10^{-3} L

6 Calculate the amount, in moles, of oxalic acid used in each titre.

n(oxalic) = 1.037×10^{-3} mol

$n(\text{oxalic}) = cV$

$= 0.05187\text{ mol L}^{-1} \times 20.0 \times 10^{-3}\text{ L}$

$= 1.037 \times 10^{-3}\text{ mol}$

7 Titrate the permanganate solution into the acidified oxalic acid until the pink colour remains for 15 s. Repeat until 3 concordant results are obtained.

Burette reading	1st titration	2nd titration	3rd titration	4th titration	5th titration
Initial reading	21.1 mL	6.6 mL	27.6 mL	1.1 mL	
Final reading	42.2 mL	27.6 mL	48.4 mL	22.0 mL	
Titre	~~21.1 mL~~	21.0 mL	20.8 mL	20.9 mL	

8 Calculate the average of the concordant titres.

$V(MnO_4^-)$ = 20.9×10^{-3} L

9 The ionic equation for this redox reaction is

$$5C_2O_4^{2-} + 2MnO_4^- + 16H^+ \longrightarrow 10CO_2 + 2Mn^{2+} + 8H_2O$$

What is the ratio of MnO_4^- to $C_2O_4^{2-}$?

$\frac{n(MnO_4^-)}{n(C_2O_4^{2-})} = \frac{2}{5}$

$n(MnO_4^-) = \frac{2}{5} n(C_2O_4^{2-})$

$n(MnO_4^-)$ = 0.4$n(C_2O_4^{2-})$

10 Calculate the amount, in moles, of permanganate reacting.

$n(MnO_4^-)$ = 4.148×10^{-4} mol

11 Calculate the concentration of the permanganate solution.

$c(MnO_4^-)$ = 0.0198 mol L^{-1}

$c(MnO_4^-) = \frac{n}{V}$

$= \frac{4.148 \times 10^{-4}\text{ mol}}{20.9 \times 10^{-3}\text{ L}}$

$= 0.0198\text{ mol L}^{-1}$

Inv 2.4 Standardising sodium thiosulfate solution

Potassium permanganate (standardised in Inv 2.2) can be used to standardise sodium thiosulfate solution by combining two redox reactions.
First, potassium permanganate oxidises iodide ions (I^-) to iodine (I_2):

$$2MnO_4^- + 16H^+ + 10I^- \longrightarrow 2Mn^{2+} + 8H_2O + 5I_2$$

then the iodine liberated is titrated against the thiosulfate solution:

$$I_2 + 2S_2O_3^{2-} \longrightarrow 2I^- + S_4O_6^{2-}$$

The endpoint is found by adding a little starch solution when the iodine has faded to pale yellow. Starch is blue-black in the presence of iodine, but turns colourless when all the iodine has reacted.

1 What is the exact concentration of the potassium permanganate solution you are using? $c(MnO_4^-) =$ 0.0200 mol L^{-1}

2 Pipette 20.0 mL samples of standard potassium permanganate solution into three conical flasks. $V(MnO_4^-) =$ 20.0×10^{-3} L

3 Calculate the amount, in moles, of permanganate used. $n(MnO_4^-) =$ 4.00×10^{-4} mol

$n(MnO_4^-) = cV$
$= 0.0200 \text{ mol L}^{-1} \times 20.0 \times 10^{-3} \text{ L}$
$= 4.00 \times 10^{-4} \text{ mol}$

4 To each flask, add about 20 mL of dilute sulfuric acid solution and about 10 mL of 10% potassium iodide solution.

5 Rinse and fill a burette with the thiosulfate solution. Slowly titrate in the thiosulfate solution to each flask, adding a few drops of starch solution when the brown colour has faded to yellow. Repeat until three concordant results are obtained.

Burette reading	1st titration	2nd titration	3rd titration	4th titration	5th titration
Initial reading	2.8 mL	22.8 mL	2.1 mL	21.8 mL	9.2 mL
Final reading	22.8 mL	42.6 mL	21.8 mL	41.5 mL	29.0 mL
Titre	20.0 mL	19.8 mL	19.7 mL	19.7 mL	19.8 mL

6 Calculate the average of concordant titres. $V(S_2O_3^{2-}) =$ 19.75×10^{-3} L

$V(S_2O_3^{2-}) = \frac{19.8 + 19.7 + 19.7 + 19.8}{4} = 19.75$ m

7 From the two equations above, what is the relationship between $n(S_2O_3^{2-})$ and $n(MnO_4^-)$? $n(S_2O_3^{2-}) =$ 5 $n(MnO_4^-)$

$\frac{n(S_2O_3^{2-})}{n(I_2)} \times \frac{n(I_2)}{n(MnO_4^-)} = \frac{\cancel{2}}{1} \times \frac{5}{\cancel{2}}$

$n(S_2O_3^{2-}) = 5\, n(MnO_4^-)$

8 Calculate the amount, in moles, of thiosulfate present. $n(S_2O_3^{2-}) =$ 2.00×10^{-3} mol

$n(S_2O_3^{2-}) = 5 \times 4.00 \times 10^{-4}$ mol
$= 2.00 \times 10^{-3}$ mol

9 Calculate the concentration of the thiosulfate solution. $c(S_2O_3^{2-}) =$ 0.101 mol L^{-1}

$c(S_2O_3^{2-}) = \frac{n}{V} = \frac{2.00 \times 10^{-3} \text{ mol}}{19.75 \times 10^{-3} \text{ L}}$
$= 0.101 \text{ mol L}^{-1}$

Inv 2.5 Standardising sodium thiosulfate using potassium iodate PowerPoint

Thiosulfate can be standardised by titrating against iodine solution. However, iodine is not easy to prepare as a primary standard because it sublimes as it is being weighed out.
To avoid this problem, we make the iodine during the reaction as follows:
A standard solution of iodate ions is reacted with excess iodide in acid conditions:

$$IO_3^- + 5I^- + 6H^+ \longrightarrow 3H_2O + 3I_2$$

The liberated iodine is then titrated against the unknown thiosulfate solution.

$$I_2 + 2S_2O_3^{2-} \longrightarrow 2I^- + S_4O_6^{2-}$$

1 Weigh out accurately about 0.71 g of dry analytical reagent grade potassium iodate, KIO_3. m(iodate) = 0.757g

2 Dissolve it in water in a 200 mL volumetric flask; shake well and make up to the mark. Again shake the solution thoroughly. Calculate the exact concentration of this solution. $c(IO_3^-)$ = 0.01769 mol L⁻¹

$c(IO_3^-) = \frac{m}{MV} = \frac{0.757\text{g}}{214.0\text{g mol}^{-1} \times 200.0 \times 10^{-3}\text{L}} = 0.01769 \text{ mol L}^{-1}$

3 Pipette 20 mL of the solution into a clean conical flask and add about 1.5 g of potassium iodide (an excess). Shake to dissolve then add 15 mL dilute H_2SO_4. $V(IO_3^-)$ = 20.0×10^{-3} L

4 Calculate the amount, in moles, of iodate used. $n(IO_3^-)$ = 3.537×10^{-4} mol

$n(IO_3^-) = cV$
$= 0.01769 \text{ mol L}^{-1} \times 20.0 \times 10^{-3}\text{ L}$
$= 3.537 \times 10^{-4}$ mol

5 Rinse and fill a burette with the thiosulfate solution. Slowly titrate the liberated iodine against the thiosulfate solution, adding a few drops of starch solution when the brown colour has faded to yellow. Repeat until three concordant results are obtained.

Burette reading	1st titration	2nd titration	3rd titration	4th titration	5th titration
Initial reading	0.3 mL	23.0 mL	1.9 mL	22.8 mL	
Final reading	23.0 mL	45.3 mL	24.4 mL	45.2 mL	
Titre	~~22.7 mL~~	22.3 mL	22.5 mL	22.4 mL	

6 Calculate the average of concordant titres. $V(S_2O_3^{2-})$ = 22.4×10^{-3} L

7 From the two equations above, what is the relationship between $n(S_2O_3^{2-})$ and $n(IO_3^-)$? $n(S_2O_3^{2-})$ = 6 $n(IO_3^-)$

$\frac{n(S_2O_3^{2-})}{n(I_2)} \times \frac{n(I_2)}{n(IO_3^-)} = \frac{2}{1} \times \frac{3}{1}$ $n(S_2O_3^{2-}) = 6 \times n(IO_3^-)$

8 Calculate the amount, in moles, of thiosulfate present. $n(S_2O_3^{2-})$ = 2.122×10^{-3} mol

$n(S_2O_3^{2-}) = 6 \times 3.537 \times 10^{-4}$ mol
$= 2.122 \times 10^{-3}$ mol

9 Calculate the concentration of the thiosulfate solution. $c(S_2O_3^{2-})$ = 0.0947 mol L⁻¹

$c(S_2O_3^{2-}) = \frac{n}{V} = \frac{2.122 \times 10^{-3}\text{ mol}}{22.4 \times 10^{-3}\text{ L}} = 0.0947 \text{ mol L}^{-1}$

Inv 2.6 Analysis of copper

The concentration of a Cu^{2+} solution can be found by reducing the Cu^{2+} to Cu^{+} with iodide, then titrating the iodine formed with thiosulfate:

$$2Cu^{2+} + 4I^{-} \longrightarrow 2CuI(s) + I_2$$
$$I_2 + 2S_2O_3^{2-} \longrightarrow 2I^{-} + S_4O_6^{2-}$$

1 Pipette 20.0 mL samples of the Cu^{2+} solution you have been given into separate conical flasks. Use a measuring cylinder to add 10 mL of 10% potassium iodide solution to each flask.

2 Rinse and fill the burette with sodium thiosulfate solution. $c(S_2O_3^{2-})$ = 0.102 mol L^{-1}

3 Titrate thiosulfate into the flask until the colour changes to a pale yellow colour. (It will be cloudy due to the formation of the white CuI precipitate.) Then add starch solution and continue to add thiosulfate until the blue colour is gone. (The blue colour will return on standing.) Repeat until concordant results are obtained.

Burette reading	1st titration	2nd titration	3rd titration	4th titration	5th titration
Initial reading	0.4 mL	12.2 mL	23.8 mL	35.3 mL	
Final reading	12.2 mL	23.8 mL	35.3 mL	46.9 mL	
Titre	~~11.8~~ mL	11.6 mL	11.5 mL	11.6 mL	

4 Average the concordant titres. $V(S_2O_3^{2-})$ = 11.57×10^{-3} L

5 Calculate the amount, in moles, of thiosulfate present. $n(S_2O_3^{2-})$ = 1.180×10^{-3} mol

$n = cV$ $n(S_2O_3^{2-}) = 0.102 \text{ mol L}^{-1} \times 11.57 \times 10^{-3} \text{ L}$
$= 1.180 \times 10^{-3}$ mol

6 Complete the expression:

$$\frac{n(Cu^{2+})}{n(I_2)} \times \frac{n(I_2)}{n(S_2O_3^{2-})} = \frac{2}{1} \times \frac{1}{2}$$

$n(Cu^{2+}) = \underline{1} \times n(S_2O_3^{2-})$

7 Calculate the amount, in moles, of Cu^{2+} in solution. $n(Cu^{2+})$ = 1.180×10^{-3} mol

8 Calculate the concentration of Cu^{2+}. $c(Cu^{2+})$ = 0.05901 mol L^{-1}

$c = \frac{n}{V}$ $c(Cu^{2+}) = \frac{1.180 \times 10^{-4} \text{ mol}}{20.0 \times 10^{-3} \text{ L}}$

9 Assume that this solution was prepared by dissolving brass in concentrated nitric acid and making it up to 1000.0 mL with water.
What mass of Cu was in the brass? (M(Cu) = 63.5 g mol^{-1}) $m(Cu^{2+})$ = 3.75 g

$n(Cu^{2+})$ in 1000.0 mL = 5.90×10^{-3} mol

$m(Cu) = nM = 0.0590 \text{ mol} \times 63.5 \text{ g mol}^{-1} = 3.75$ g

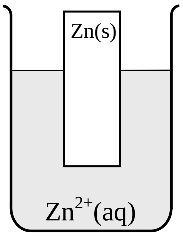
10 Calculate the percentage of copper in the brass if it had a mass of 5.70 g. $\%Cu = \frac{m(Cu)}{m(brass)} \times \frac{100}{1} = \frac{3.75 \text{ g}}{5.70 \text{ g}} \times \frac{100}{1} = 65.7\%$ %Cu = 65.7%

Inv 3.1 Electrochemical cells 1 PowerPoint

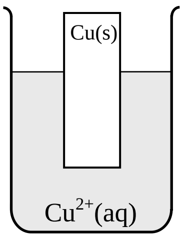
All redox reactions involve electron transfer. This can happen even when the two reactants are physically separated, say, electrons transferring from one to another through a wire. This is what happens in an electrochemical cell.

1 Clean the surface of the metal strips you use.

2 Prepare a Zn^{2+}/Zn half cell by placing a strip of zinc into a beaker of zinc sulfate solution so that the solution covers three-quarters of the zinc strip.



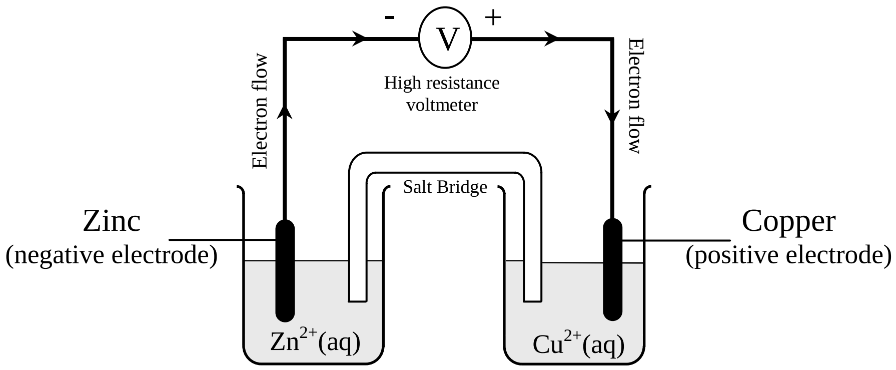
3 Prepare a Cu^{2+}/Cu half cell in a similar manner using a copper strip and copper sulfate.



4 Connect a wire from the zinc electrode (strip) to the negative terminal of a multimeter set on voltage. Record the reading on the meter.___0.00 V___

5 Connect a wire from the copper electrode (strip) to the positive terminal of the meter. Record the reading on the meter.___0.00 V___

6 Place the half cells next to each other and connect them via a salt bridge. Record the reading on the meter.___1.12 V___



7 Which way do electrons flow in the circuit? ___Left to right (from Zn to Cu)___

8 What is the cathode? ___RHE (copper)___

9 What is the anode? ___LHE (Zinc)___

10 Write the half equation for the reaction occurring in the Zn^{2+}/Zn half cell.

$Zn(s) \rightarrow Zn^{2+}(aq) + 2e^-$

11 Is this oxidation or reduction? ___Oxidation___

12 Write the half equation for the reaction occurring in the Cu^{2+}/Cu half cell.

$Cu^{2+}(aq) + 2e^- \longrightarrow Cu(s)$

13 Is this oxidation or reduction? reduction

14 Combine your half equations from **11** and **13** above to give the overall cell reaction.

$Zn(s) + Cu^{2+}(aq) \longrightarrow Zn^{2+}(aq) + Cu(s)$

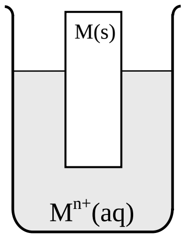
15 What is the EMF of the above cell? 1.12 V

Inv 3.2 Electrochemical cells 2

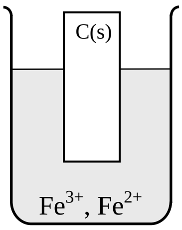
We can use the electrode potentials of redox systems to determine the relative strengths of oxidants and reductants. Your teacher will give you a range of oxidants and reductants from which you can prepare different half-cells. Electrochemical cells are made by combining two half-cells.

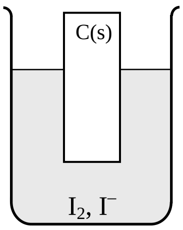
1 Clean the surface of the metal strips you use.

2 Prepare M^{n+}/M half cells by placing a strip of metal into a beaker of the metal sulfate solution so that the solution covers three-quarters of the metal strip.

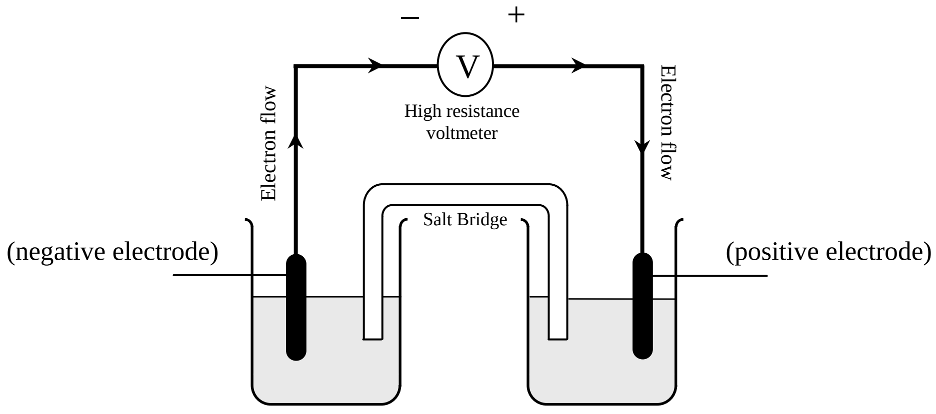


3 Prepare a Fe^{3+}/Fe^{2+} half cell using a carbon electrode in equal volumes of iron(II) sulfate and iron(III) nitrate and an I_2/I^- half cell using a carbon electrode in equal volumes of iodine solution and potassium iodide solution.





4 Prepare cells by combining two half cells, connecting the two electrodes with a voltmeter and the solutions with a salt bridge.



5 For each cell measure the voltage (EMF) and record it in the table shown along with the direction of electron flow and the oxidant and reductant for each cell.

Cell	Cell voltage		Electron flow			
	measured	calculate	from	to	Oxidant	Reductant
$Zn/Zn^{2+}//Cu^{2+}/Cu$	1.11 V	1.1 V	LHE	RHE	Cu^{2+}	Zn
$Mg/Mg^{2+}//Zn^{2+}/Zn$	0.85 V	1.6 V	LHE	RHE	Zn^{2+}	Mg
$Mg/Mg^{2+}//Cu^{2+}/Cu$	1.98 V	2.7 V	LHE	RHE	Cu^{2+}	Mg
$Zn/Zn^{2+}//Fe^{3+},Fe^{2+}/C$	1.49 V	1.53 V	LHE	RHE	Fe^{3+}	Zn
$Zn/Zn^{2+}//I_2,I^-/C$	1.10 V	1.38 V	LHE	RHE	I_2	Zn

6 Calculate the cell voltages from $E°$ values from data tables and compare with the measured values. Suggest any reasons for differences.

The values will be different from standard values if the conditions were not standard - including carbon electrodes instead of platinum ones.

7 Arrange the oxidants in order of increasing ability to be reduced.

$Zn^{2+} < Cu^{2+} < I_2 < Fe^{3+}$

Inv 3.3 The lead-acid cell PowerPoint

One of the most useful practical electrochemical cells is the lead-acid cell used in car batteries. It is so useful because its electrode reactions can be reversed by passing an electric current back through the cell.

1 Clean the surface of two lead plates and place them in a 250 mL beaker. Add about 150 mL of about 2 mol L^{-1} of sulfuric acid.

2 Connect a voltmeter between the plates and record the voltage.

Voltage reading: 0.0 V

3 Remove the voltmeter and replace it with a DC power supply. Adjust the supply to about 3 V, switch on and wait and observe for about 10 minutes. Note any changes that take place.

Observations: Bubbles of gas are seen at the negative electrode, while the positive electrode is coated in a very dark brown substance.

4 Replace the DC supply with the voltmeter and measure the voltage. (Make sure of the polarity.)

Voltage reading: 1.99 V

5 Replace the voltmeter with a 1.5 V bulb and observe.

Observation: The bulb lights up.

During the charging of the cell the lead, Pb(s), of the cathode is converted to lead (IV) oxide, PbO_2(s).

6 Write equations for the reactions that take place at each electrode during discharge.

Anode: $Pb(s) \longrightarrow Pb^{2+}(aq) + 2e^-$

Cathode: $PbO_2(s) + 4H^+(aq) + 2e^- \longrightarrow Pb^{2+}(aq) + 2H_2O(l)$

Inv 4.1 Heat and phase changes

When a substance changes state from one phase to another the temperature remains the same, but heat is still absorbed or given out in the process.

1 Weigh a clean, dry 250 mL beaker. m_1 = 96.85 g

2 Add about 200 mL of hot water from the tap (about 55 °C) and reweigh. m_2 = 290.9 g

Initial temperature is: T_1 = 39°C

The mass of the water added is: $m_{Water} = m_2 - m_1$ = 200.05 g

3 Add about 80 g of ice, stir and note the temperature when all of the ice has melted. T_2 = 13°C

4 Reweigh the beaker and contents to find the mass of ice that was added. m_3 = 353.2 g

The mass of the ice added is: $m_{Ice} = m_3 - m_2$ = 56.3 g

The change in temperature of the water is: $\Delta T_1 = T_2 - T_1$ = 26 °C

The change in temperature of the ice is: $\Delta T_2 = T_2 - 0°C$ = 13°C

Calculations: The heat lost by the water cooling from its original temperature to the final temperature is equal to the heat gained by the ice as it melts, plus that gained by the melted ice raising from 0°C to the final temperature.

$$m_{Water} \times \text{specific heat} \times \Delta T_1 = m_{Ice} \times \text{latent heat} + m_{Ice} \times \text{specific heat} \times \Delta T_2$$

Heat lost by water: 200.05 g × 4.2 × 26°C = 21845 J (1)

Heat gained by melted ice warming: 56.3 g × 4.2 × 13°C = 3074 J (2)

The difference is the energy required to melt the ice: (1) – (2) = 18771 J

This is the heat needed to melt m_{Ice} i.e. 56.3 g g of ice.

5 Calculate the amount of heat needed to melt 1.00 mol of ice.

$n(\text{ice}) = \frac{m}{M} = \frac{56.3\text{g}}{18\text{g mol}^{-1}} \doteq 3.128 \text{ mol}$

$\Delta_{fus}H(\text{ice}) = \frac{18771 \text{ J}}{n(\text{ice})} = \frac{18771 \text{ J}}{3.128 \text{ mol}} = 6.0 \text{ kJ mol}^{-1}$

6 How do your results compare with those of other groups and latent heat from data tables?

The data book value for $\Delta_{fus}H(\text{ice}) = 6.0 \text{ kJ mol}^{-1}$, which is equal to this experimental value. Other groups got very similar values.

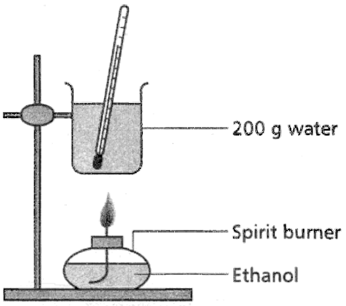
Inv 4.2 Heats of combustion

The heat of combustion of a substance is the quantity of heat liberated when one mole of that substance is burnt completely in air (oxygen). This is of value in determining such things as the relative merits of various fuels and in calculating other quantities such as heats of formation.

An experiment to compare the energy output of different alcohols can be performed by using the apparatus shown.

The alcohols to be tested are burnt in a small spirit burner.

With such simple equipment it is not possible to absorb all the heat produced, only a fraction of it. Hence, everything must be kept the same, except the fuel, to ensure this fraction is the same for each of the alcohols tested.



1 Accurately measure 200 g of tap water into the 250 mL beaker (or conical flask). Clamp in position and record the initial temperature of the water to the nearest 0.2 °C in Table 1.

2 Place approximately 10 mL of methanol in the spirit burner, replace the top, weigh the burner and contents, and record this mass in Table 1.

3 Place the burner under the centre of the flask, shield it from drafts and light the burner. Stir the water with the thermometer and when the temperature has risen 10 °C, extinguish the burner.

4 Continue stirring for 10 seconds, then record the temperature in Table 1.

5 Re-weigh the burner and record its mass in Table 1.

6 Empty the beaker (or flask) and rinse it with tap water to cool it to room temperature. Discard the remaining methanol, and sponge the methanol from the lower part of the wick with filter paper. (Do not disturb the wick protruding through the top.) Now repeat steps **1– 6** for each of the other alcohols.

Table 1

Alcohol	**Temperature of water (°C)**			**Energy absorbed (kJ)**	**Mass of burner and alcohol (g)**		
	initial	**final**	**change**		**initial**	**final**	**loss**
Methanol	22.4	33.6	11.2	9.363	15.6	14.75	0.86
Ethanol	22.8	33.8	11.0	9.196	16.15	15.50	0.65
Propan-1-ol	23.0	34.0	11.0	9.196	16.85	16.27	0.58
Butan-1-ol	23.6	33.8	10.2	8.527	17.54	17.05	0.49

7 Calculate the change in temperature of the water, and hence calculate the energy absorbed by the water in kJ, assuming that 1.0 mL of water requires 4.18 J to raise its temperature by 1 °C.

8 Calculate the mass of each alcohol burnt and record this in column 8 of Table 1, then calculate the amount, in moles, of each alcohol burnt and record this in column 4 of Table 2.

9 Use your values for column 5 of Table 1 and column 4 of Table 2 to calculate the apparent $\Delta_c H$ for each alcohol (apparent, because we know only a fraction of the energy produced has been absorbed by the water).

Table 2

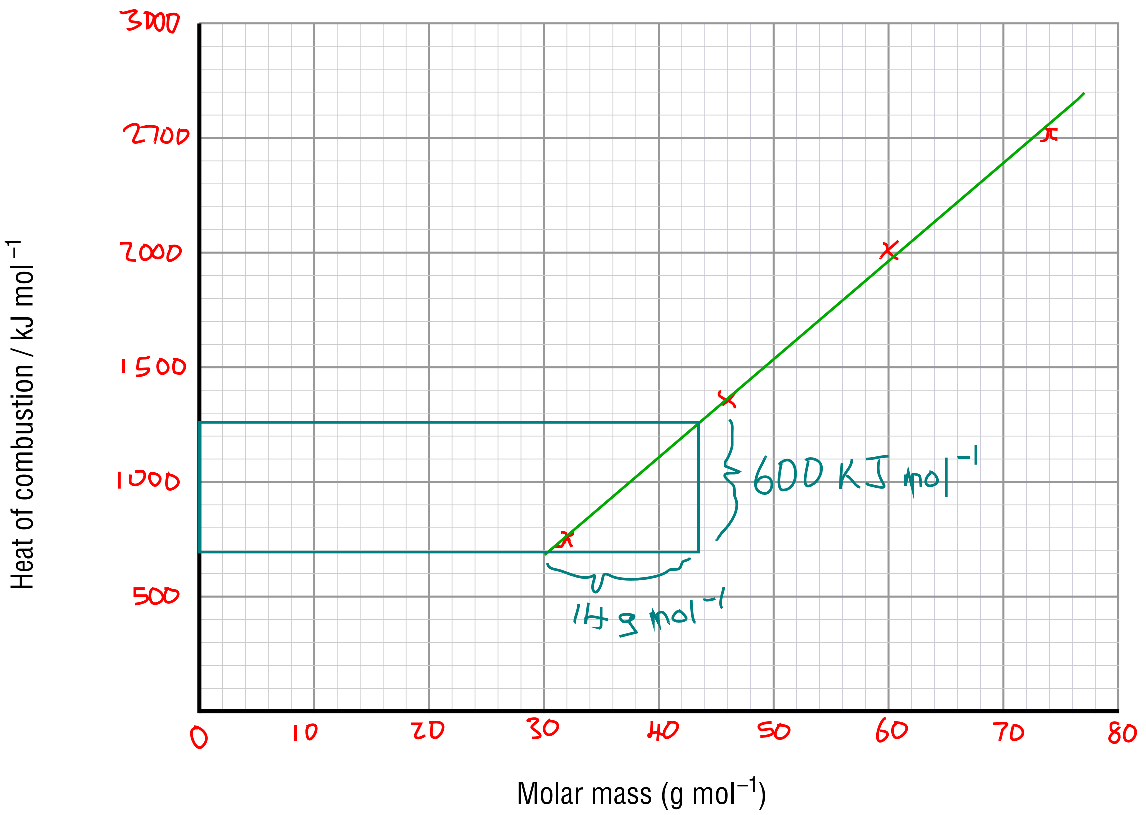
Alcohol	**Molecular formula**	**Molar mass ($g\ mol^{-1}$)**	**Amount burnt (mol)**	**Apparent $\Delta_c H$ ($kJ\ mol^{-1}$)**	**Adjusted $\Delta_c H$ ($kJ\ mol^{-1}$)**	**Book $\Delta_c H$ ($kJ\ mol^{-1}$)**
Methanol	CH_3OH	32	0.0269	348	732	715
Ethanol	C_2H_5OH	46	0.0141	652	1372	1372
Propan-1-ol	C_3H_7OH	60	9.67×10^{-3}	951	2000	2012
Butan-1-ol	C_4H_9OH	74	6.62×10^{-3}	1288	2710	2670

10 The book value for $\Delta_c H$(ethanol) is 1372 kJ mol^{-1}. Use your apparent $\Delta_c H$(ethanol) to calculate the fraction of energy produced that was absorbed by the water.

$$\text{Fraction absorbed} = \frac{\Delta_c H(\text{book})}{\Delta_c H(\text{apparent})} = \frac{1372}{651} = 2.104$$

11 Multiply your apparent $\Delta_c H$ by the fraction calculated in **10** to find the adjusted $\Delta_c H$ for each of the other alcohols.

12 Plot a graph of molar mass versus heat of combustion for these alcohols, using your adjusted values. Draw the line of best.



13 Comment on the shape of the graph and the comparison between your values and the book values.

The difference between adjacent molecules is $-CH_2-$. From the graph a difference of 14 g mol^{-1} (CH_2) is equal to about 600 KJ mol^{-1}! The experimental values are close to the book values.

Inv 4.3 Finding the enthalpy change for redox reactions

The heat of reaction when one metal displaces another from a solution of its ions can be measured and compared to the $E°$ value for the same cell reaction.

Note: wash your hands after handling metal powders – they can cause eye damage.

1 Use a measuring cylinder to carefully measure out 25 mL of 0.4 mol L^{-1} copper(II) sulfate solution into a clean polystyrene cup jacketed inside another polystyrene cup.

2 Measure the starting temperature to 0.5 °C t_s = 14.5 °C

3 Add 1 g (an excess) of zinc powder, and stir thoroughly with a glass rod.

4 Record the peak temperature. t_f = 34.5 °C

5 Determine the rise in temperature Δt = 20 °C

6 The equation for the reaction can be written as:

$$Cu^{2+}(aq) + Zn(s) \longrightarrow Cu(s) + Zn^{2+}(aq)$$

7 Calculate the number of moles of Cu^{2+} used in this reaction. n(Cu) = 0.010 mol

$n(Cu^{2+}) = cV = 0.4\ mol\ L^{-1} \times 25 \times 10^{-3}\ L$

$= 0.010\ mol$

8 Use the following formula to determine the amount of heat released in the reaction:

Heat = mass × specific heat × temperature change

(where specific heat capacity of water = 4.2 J g^{-1} $°C^{-1}$ and 1 mL water = 1 g) heat = 2100 J

heat = 25 g × 4.2 J g^{-1} $°C^{-1}$ × 20 °C = 2100 J

9 Calculate the heat released, ΔH, per mole of Cu ΔH = 210 kJ mol^{-1}

$\Delta H = \dfrac{2100\ J}{0.010\ mol} = 210000\ J\ mol^{-1}$

$= 210\ kJ\ mol^{-1}$

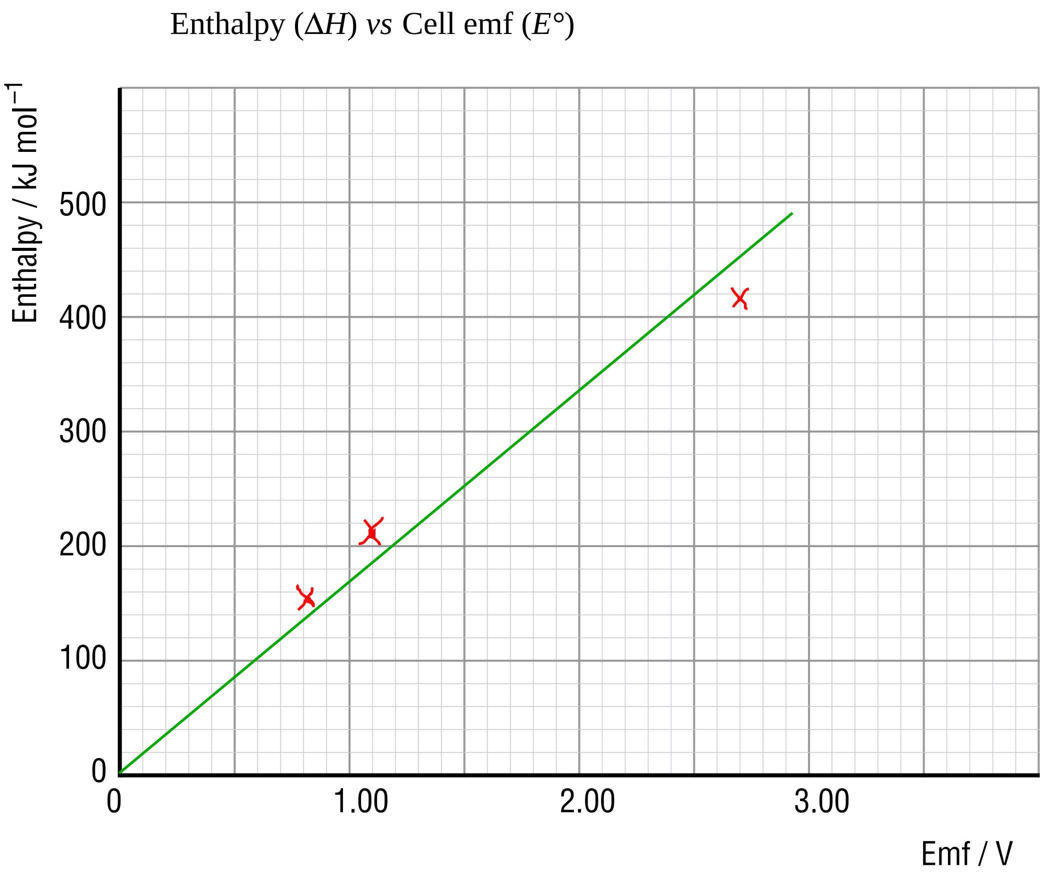
10 Repeat the above experiment using (a) iron powder and (b) magnesium powder or turnings. (For Mg, 0.5 g should be sufficient.)

Cell reaction	***E°*(LHE)**	***E°*(RHE)**	***E°*$_{cell}$**	**Δ*H***
Zn(s) / Zn^{2+}(aq) // Cu^{2+}(aq) / Cu(s)	–0.76 V	+0.34 V	1.1 V	210 kJ mol^{-1}
Fe(s) / Fe^{2+}(aq) // Cu^{2+}(aq) / Cu(s)	–0.47 V	+0.34 V	0.81 V	155 kJ mol^{-1}
Mg(s) / Mg^{2+}(aq) // Cu^{2+}(aq) / Cu(s)	–2.36 V	+0.34 V	2.70 V	415 kJ mol^{-1}

11 Calculate the $E°_{cell}$ for each reaction using the formula

$$E°_{cell} = E°_{RHE} – E°_{LHE}$$

12 Plot the ΔH values against the $E°_{cell}$ for the three cell reactions. The plot should go through the origin.



13 Is there a relationship between the energy released as heat, and energy released as electricity?

Yes - there is a linear relationship between them.

14 What would happen if the metals were partly oxidised before the reactions? Explain.

If the metals were partly oxidised before the reaction the heat given off would be less, ie the temperature rise would be less because there would be less material to react. This seems to have happened with the magnesium.

Inv 4.4 Hess's law: the law of heat summation

The heat of a reaction is determined only by the difference in enthalpy between the reactants and products and is independent of the path of the reaction and the manner by which it is brought about. This is illustrated by the formation of sodium chloride by two different reaction paths.

A The two-stage path:

1. The reaction between solid sodium hydroxide and water

$$NaOH(s) + H_2O(l) \longrightarrow Na^+(aq) + OH^-(aq) \quad \Delta_r H_1$$

2. The reaction between sodium hydroxide solution and dilute hydrochloric acid

$$Na^+(aq) + OH^-(aq) + H^+(aq) + Cl^-(aq) \longrightarrow H_2O(l) + Na^+(aq) + Cl^-(aq) \quad \Delta_r H_2$$

B The single-stage path:

The reaction between solid sodium hydroxide and dilute hydrochloric acid

$$NaOH(s) + H^+(aq) + Cl^-(aq) \longrightarrow H_2O(l) + Na^+(aq) + Cl^-(aq) \quad \Delta_r H_3$$

If Hess's law is correct then, within experimental error,

$$\Delta_r H_1 + \Delta_r H_2 = \Delta_r H_3$$

$\Delta_r H_1$ heat of reaction for dissolution of sodium hydroxide

1 Place one styrofoam cup inside another one to form a calorimeter.

2 Place 100 mL of water in the styrofoam cup and note the temperature. T_1 = 14.8 °C

3 Weigh out rapidly 0.05 mol (2 g) of sodium hydroxide pellets (corrosive) to the nearest 0.1 g (about 20 pellets). m = 2.1 g

4 Calculate the amount of sodium hydroxide used. n(NaOH) = 0.0525 mol

$n(NaOH) = \frac{m}{M} = \frac{2.1\text{ g}}{40\text{ g mol}^{-1}} =$

5 Add the solid sodium hydroxide to the water, stir gently with a thermometer and record the highest temperature. T_2 = 20.3 °C

6 Determine the rise in temperature. ΔT = 5.5 °C

7 Heat energy (ΔH_1) = mass of water × temperature rise × 4.2 ΔH_1 = 2310 J

Heat = 100 g × 5.5 °C × 4.2 J g^{-1} °C^{-1} = 2310 J

8 Calculate the heat energy in joules for 1 mole of NaOH $\Delta_r H_1$ = 44.0 kJ mol^{-1}

$\Delta H_1 = \frac{\text{Heat}}{n} = \frac{2310\text{ J}}{0.0525\text{ mol}} = 44000\text{ J}$

$\Delta_r H_2$ heat of reaction of sodium hydroxide solution with hydrochloric acid

9 Rinse the styrofoam cup and add 50 mL of 1 mol L^{-1} NaOH(aq) (0.05 mol). Measure out 50 mL of 1 mol L^{-1} HCl(aq) (0.05 mol) into a measuring cylinder and record both temperatures. The initial temperature is the mean of the two. T_1 = 16.75 °C

10 Add the acid to the hydroxide solution, stir gently with a thermometer and record the highest temperature. T_2 = 24.5 °C

11 Determine the rise in temperature. ΔT = 7.75 °C

12 Calculate the amount of sodium hydroxide used in the reaction. $n = cV = 1 \text{ mol L}^{-1} \times 50 \times 10^{-3} \text{ L}$ $= 0.05 \text{ mol}$ $n(NaOH) =$ 0.05 mol

13 Calculate the heat energy released in the reaction:
Heat energy (ΔH_2) = mass of solution × temperature rise × 4.2 $\Delta H_2 =$ 3255 J
heat $= (50 + 50)\text{g} \times 7.75\,°\text{C} \times 4.2 \text{ J g}^{-1}\,°\text{C}^{-1} = 3255 \text{ J}$

14 Calculate the heat energy in joules for 1 mole of reactants $\Delta_r H_2 =$ 65 kJ mol^{-1}
$\Delta_r H_2 = \frac{\text{heat}}{n} = \frac{3255 \text{ J}}{0.05 \text{ mol}} = 65\,100 \text{ J mol}^{-1}$

$\Delta_r H_3$ heat of reaction between solid sodium hydroxide and hydrochloric acid

11 Rinse the styrofoam cup and add 100 mL of 0.5 mol L^{-1} HCl(aq) (0.05 mol) and record initial temperature. $T_1 =$ 17.0 °C

12 Weigh out rapidly 0.05 mol (2 g) of sodium hydroxide pellets (corrosive) to the nearest 0.1 g (about 20 pellets) as in **3**. $m =$ 2.0 g

13 Add the sodium hydroxide to the styrofoam cup and stir gently with a thermometer and record the highest temperature. $T_2 =$ 29.5 °C

14 Determine the rise in temperature. $\Delta T =$ 12.5 °C

15 Calculate the amount of sodium hydroxide used in the reaction. $n(NaOH) = \frac{m}{M} = \frac{2.0 \text{ g}}{40 \text{ g mol}^{-1}} = 0.050 \text{ mol}$ $n(NaOH) =$ 0.050 mol

16 Calculate the heat energy released in the reaction:
Heat energy (ΔH_3) = mass of solution × temperature rise × 4.2 $\Delta H_3 =$ 5250 J
heat $= 100 \text{ g} \times 12.5\,°\text{C} \times 4.2 \text{ J g}^{-1}\,°\text{C}^{-1} = 5250 \text{ J}$

17 Calculate the heat energy in joules for 1 mole of reactants $\Delta_r H_3 =$ 105 kJ mol^{-1}
$\Delta_r H_3 = \frac{\text{heat}}{n} = \frac{5250 \text{ J}}{0.050 \text{ mol}} = 105\,000 \text{ J mol}^{-1}$

Comparing $\Delta_r H_1 + \Delta_r H_2$ to $\Delta_r H_3$

18 How does the value of $\Delta_r H_1 + \Delta_r H_2$ compare to the value of $\Delta_r H_3$, considering possible sources of experimental error and their magnitude? Draw a conclusion.

$\Delta_r H_1 + \Delta_r H_2 =$ 44 kJ mol^{-1} + 65 kJ mol^{-1} = 109 kJ mol^{-1}

$\Delta_r H_3 =$ 105 kJ mol^{-1}

Both values lie in the range 107 kJ mol^{-1} ± 2% which suggests the law is valid. The weighing errors and accuracy of measuring cylinders, and solution concentration are of this order of accuracy.

Inv 5.1 Transition metal chemistry 1: Manganese

Transition metals can have a number of different oxidation states and their compounds and complexes are almost always coloured.

1 Manganese (II) sulfate and potassium permanganate are common laboratory reagents. What are their colours and what are the oxidation states of manganese in each compound?

$MnSO_4$, Manganese (II) is pale pink

MnO_4^-, manganese (VII) is purple.

2 Add a little dilute NaOH(aq) to about 2 mL of $MnSO_4$(aq) in a test tube. What do you observe?

A pale pink - buff coloured precipitate.

Filter and allow the precipitate to stand in air. What do you observe?

The colour of the precipitate goes darker brown.

3 Write an ionic equation for the reaction that occurs in **2**.

$Mn^{2+}(aq) + 2OH^-(aq) \longrightarrow Mn(OH)_2(s)$

4 MnO(OH) is the product formed in 3. Write an ion-electron half-equation for its formation.

Alkaline conditions

$Mn(OH)_2(s) + OH^-(aq) \longrightarrow MnO(OH)(s) + H_2O(l) + e^-$

5 What is the oxidation state for manganese in MnO(OH)? +3

6 Add a few drops of $KMnO_4$(aq) to $MnSO_4$(aq) in a test tube. What do you observe?

A brown precipitate forms.

7 The product formed in **6** is MnO_2. Write an ion-electron half-equation for its formation from MnO_4^-.

$MnO_4^-(aq) + 4H^+ + 3e^- \longrightarrow MnO_2(s) + 2H_2O(l)$

8 What is the oxidation state for manganese in MnO_2? +4

9 Add 3 drops of $KMnO_4$(aq) to each of two test tubes containing a few mL of 30% NaOH(aq). Then add 1 drop of 1 mol L^{-1} $NaHSO_3$(aq) to one test tube and 5 drops to the other. Carefully shake the test tubes and leave to stand for a couple of minutes. What do you observe?

1 drop A blue solution forms

5 drops A green solution forms

10 MnO_4^{3-}(aq) is blue and MnO_4^{2-}(aq) is green. Write ion-electron half-equations for their formation in **9**.

$MnO_4^- + 2e^- \longrightarrow MnO_4^{3-}$

$MnO_4^- + e^- \longrightarrow MnO_4^{2-}$

11 What are the oxidaton states for the two manganese species formed in **9**?

$MnO_4^{3-} = +5$ $MnO_4^{2-} = +6$

Inv 5.2 Transition metal chemistry 2: Vanadium

Transition metals can have a number of different oxidation states and their compounds and complexes are almost always coloured. Vanadium has the following: violet [V(II)], V^{2+}; green [V(III)], V^{3+}; blue [V(VI)], VO^{2+}; yellow/orange [V(V)], VO_2^+.

1 Put half a spatula of solid ammonium vanadate, NH_4VO_3, (TOXIC) into a conical flask, then add about 20 mL of dilute sulfuric acid (2 mol L^{-1}). Swirl the liquid to dissolve the solid.

Colour of the solution: Yellow   Oxidation state of vanadium: +5

2 Decant off the liquid from 1 into a clean conical flask, add 20 mL of sulfuric acid (2 mol L^{-1}) and a spatula of zinc powder. Place some cotton wool loosely in the top of the flask and swirl the flask gently. Note the colour changes that occur in the mixture.

Colour changes: yellow to green to blue to green to purple (violet)

3 Leave the flask with the cotton wool plug until the final colour is violet.

Colour: violet   Oxidation state of vanadium: +2

4 Pour 10 mL of this solution into each of two 100 mL beakers. Leave one beaker exposed to the air, and the other other add about 10 mL dilute nitric acid (1–2 mol L^{-1}) and swirl the mixture. Note the colour changes in each beaker.

a In air — colour change green   Oxidation state of vanadium +3

b In HNO_3 — colour change blue   Oxidation state of vanadium +4

5 **a** Write ion-electron half-equation for the conversion of VO_2^+ to VO^{2+}.

$VO_2^+(aq) + 2H^+(aq) + e^- \longrightarrow VO^{2+}(aq) + H_2O(l)$

b What would you see during this reaction?

The yellow solution would turn blue.

6 **a** Write ion-electron half-equation for the conversion of V^{3+} to VO^{2+}.

$V^{3+}(aq) + H_2O \longrightarrow VO^{2+}(aq) + 2H^+ + e^-$

b What would you see during this reaction?

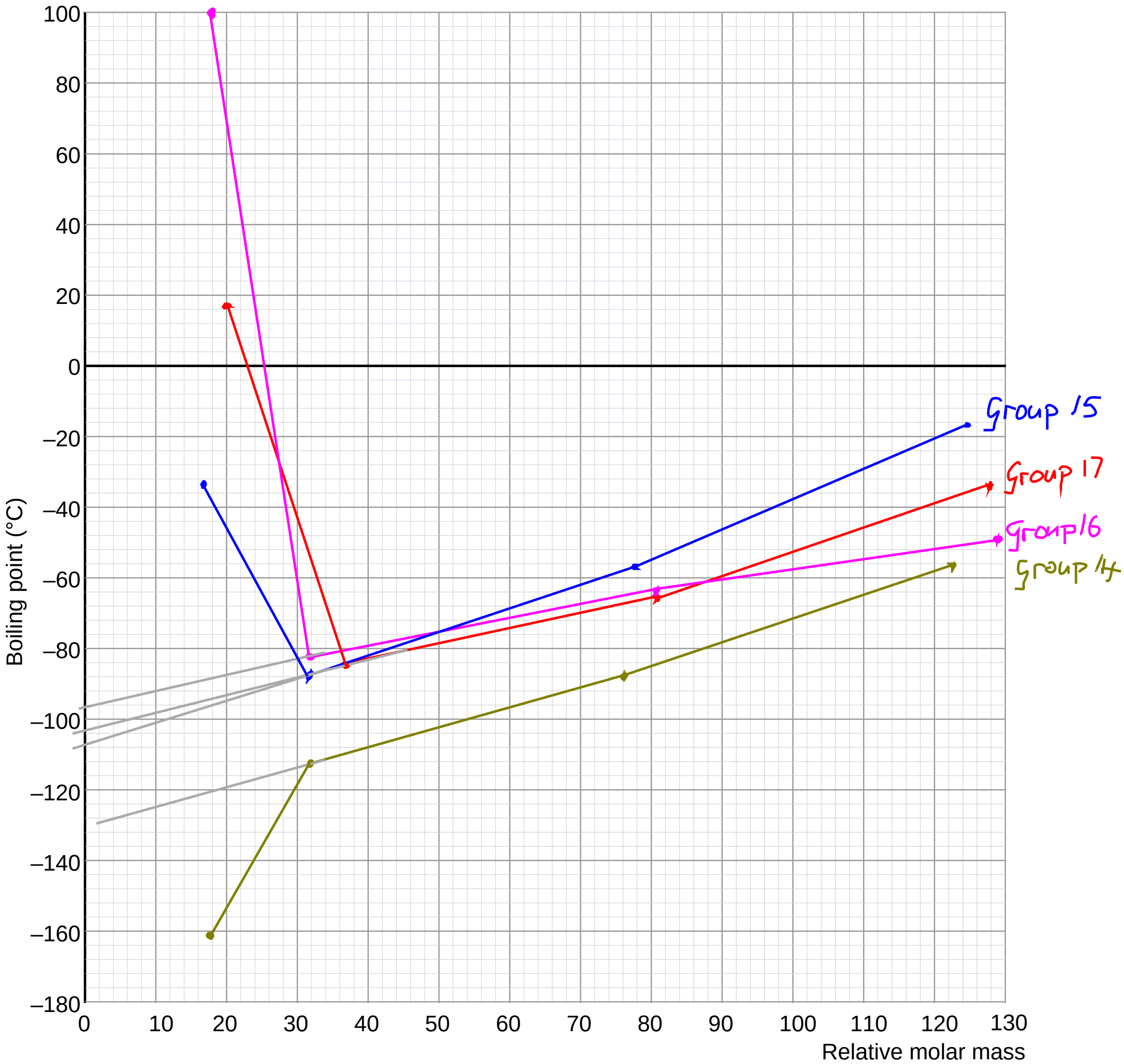
The green solution would turn blue.

Inv 6.1 Hydrogen bonding

Some compounds do not follow the same trend in properties, such as boiling point, as do other members of their group. This is due to a very strong dipole-dipole force called hydrogen bonding.

Group 17	M_r	BP °C	Group 16	M_r	BP °C	Group 15	M_r	BP °C	Group 14	M_r	BP °C
HF	20	(93)	H_2O	18	(-84)	NH_3	17	(−96)	CH_4	16	(-122)
HCl	36.5	−85	H_2S	34	−83	PH_3	34	−88	SiH_4	32	−112
HBr	81	−67	H_2Se	81	−66	AsH_3	78	−57	GeH_4	76.6	−88
HI	128	−35	H_2Te	129.6	−49	SbH_3	125	−17	SnH_4	122.7	−52

1 On the grid following draw graphs of the boiling points of the 4 groups against their relative molar masses, using just the data above. Use a different colour for each group.



2 What trend do you observe?

As molar mass increases the boiling point increases.

3 Explain this trend.

The increase in boiling point happens because the molecules are getting larger, with more electrons, and so the intermolecular forces between instantaneous dipoles and induced dipoles are stronger.

4 Using the trends and the graphs you have drawn, what do you think the missing boiling points in the table should be? Write your estimations in the table in pencil.

5 The actual boiling points are: HF (19 °C), H_2O (100 °C), NH_3 (–33 °C), CH_4 (–162 °C). Add these points to the graphs you have drawn. What do you observe?

The boiling points of HF, H_2O and NH_3 are very much higher than the trend suggests, while CH_4 has a lower BP than the trend suggests.

When hydrogen is bonded to a very electronegative element – fluorine, oxygen or nitrogen – the bond formed is highly polar; the hydrogen is almost a bare proton. Consequently, the hydrogen of one molecule exerts a strong electrostatic attraction for the very electronegative atom in a neighbouring molecule. This inter-molecular bond is called a "hydrogen bond" and the boiling point is thus higher than would be expected.

Inv 7.1 Structures of organic molecules

We use molecular models to help us understand the structures, geometries and properties of molecules. In most model sets black represents carbon, white is used for hydrogen, red for oxygen and green for chlorine.

1 Build a model of CH_2Cl_2.

a What is this compound called? dichloromethane

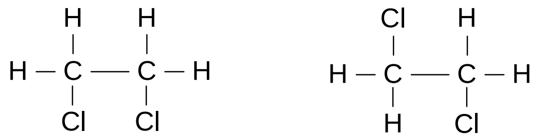
b Is this compound polar? Explain your answer.
Yes. The C–Cl bond is polar because Cl is more e⁻neg than C, and the shape of the molecule puts both dipoles on the same side of the carbon.

2 Rebuild your molecule putting the chlorines in different places.

Is this the same compound or different? Same

3 Make a model of ethane, CH_3CH_3. Firmly hold one of the carbon atoms. Now try to rotate the other CH_3 group about the C–C bond. Is rotation possible? Yes

4 Build each of the molecules shown on the right.

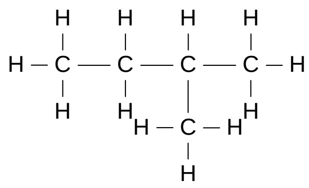


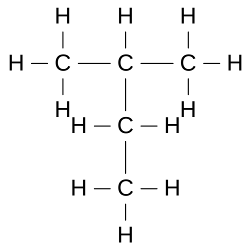
a Are they identical or are they isomers?
Identical

Explain why. Rotation about the C–C bond is possible.

b Is the above compound polar or non-polar? Non-polar

5 Build each of these molecules in turn:





Are the two compounds identical?

Yes

6 Build a model of ethene, CH_2CH_2.

a Is this a planar molecule? Yes

b Is it possible to rotate one end of the molecule as with ethane? No

c What is the effect of double bonds on rotation? A double bond prevents rotation

7 Replace two hydrogens with chlorines.

How many *isomeric* molecules can you make of $C_2H_2Cl_2$? 3

8 Two of these isomers are cis -1,2-dichloroethene and trans-1,2-dichloroethene. Build both models.

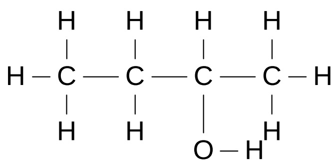
a Why are these two compounds not identical? The double bond prevents rotation,

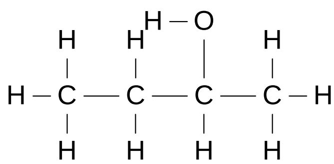
b Which isomer is polar: *cis*- or *trans*-1,2-dichloroethene? cis 1,2-dichloroethene

9 The *cis* and *trans* forms are called geometrical isomers of each other. Build a third isomeric $C_2H_2Cl_2$ which is related to the above two by structural isomerism.

What is its name? 1,1 dichloroethene

10 Build a model of butan-2-ol, $CH_3CH_2CHOHCH_3$. Compare your model with those made by other students. You should find that there are two distinct groups.





These two different molecules are OPTICAL ISOMERS. What is it about these models that shows that there are two different compounds present?

Although the atoms are joined to the same atoms in each version, the two isomers are mirror images of each other and cannot be super-imposed.

Inv 8.1 Oxidation of alcohols PowerPoint

Primary (1°), secondary (2°) and tertiary (3°) alcohols react differently with oxidising agents. These oxidation reactions can be used to distinguish between the different types of alcohols.

1 Mix 1 mL of ethanol with 5 drops of potassium dichromate solution and 10 drops of dilute sulphuric acid in a test tube. Place the test tube in a beaker of hot water to warm the mixture. What do you observe? Note the smell.

The colour of the mixture changes from orange to green/blue.
The smell of ethanol changes (now a cherry brandy-type smell).

2 Repeat **1** using 1 mL of propan-2-ol instead of ethanol. What do you observe? Note the smell.

The colour of the mixture changes from orange to green/blue.
The smell changes (now a nail-polish remover type smell).

3 Repeat **1** this time using 2-methylpropan-2-ol (tertiary butyl alcohol). What do you observe?

No visible sign of reaction: both colour and smell unchanged

4 Repeat **1** to **3** using potassium permanganate solution instead of potassium dichromate solution. What do you observe?

i The purple colour changes to colourless and smell changes as above.

ii The purple colour changes to colourless and smell changes as above.

iii No change in colour or smell.

A **primary alcohol** is oxidised to an aldehyde (alkanal) and eventually to a carboxylic acid. A **secondary alcohol** is oxidised to the corresponding ketone (alkanone). **Tertiary alcohols** do not undergo oxidation under the above conditions.

5 Write equations giving the organic reactants and products for the reactions that have taken place.

1 : $CH_3CH_2OH \xrightarrow{[O]} CH_3CHO \; (\xrightarrow{[O]} CH_3COOH)$

2 : $CH_3CHOHCH_3 \xrightarrow{[O]} CH_3-C(=O)-CH_3$

6 Give the name and colour of the oxidising species in each case and the name and colour of the species to which they were reduced.

1-3 $Cr_2O_7^{2-}$ orange is reduced to Cr^{3+} blue/green
dichromate chromium (III)

4 MnO_4^- purple is reduced to Mn^{2+} colourless
permanganate manganese (II)

Inv 8.2 Reactions of alcohols with Lucas reagent PowerPoint

A test that can be used to distinguish between the three types of alcohol is the Lucas test.

1 Put about 1 mL of ethanol in a dry test tube. Add 10 drops of Lucas reagent (a mixture of concentrated hydrochloric acid (corrosive) and anhydrous zinc chloride) and shake to mix well. Stand the test tube in a test tube rack. What do you observe?

No visible change

2 Repeat **1** using 1 mL of propan-2-ol instead of ethanol. What do you observe?

The mixture goes cloudy after a few minutes

3 Repeat **1**, this time using 2-methylpropan-2-ol (tertiary butyl alcohol). What do you observe?

The mixture goes cloudy almost immediately.

Leave any unreacted test tubes in the rack until the end of the lesson.

4 List the alcohols in order of their rate of reaction with the Lucas' reagent. Put the fastest first.

2-methylpropan-2-ol (3°) > propan-2-ol (2°) > ethanol (1°)

5 Put the alcohols in order of increasing strength of the R- O bond.

ethanol (1°) > propan-2-ol (2°) > 2-methylpropan-2-ol (3°)

6 Write equations for reaction of each alcohol with concentrated hydrochloric acid (the zinc chloride is a catalyst). Use structural formulae and in each case name both products and reactants.

$$CH_3-CH_2-OH + HCl \xrightarrow{ZnCl_2} \text{no reaction}$$

ethanol

$$CH_3-CH(OH)-CH_3 + HCl \xrightarrow{ZnCl_2} CH_3-CH(Cl)-CH_3 + H_2O$$

propan-2-ol → 2-chloropropane

$$CH_3-C(CH_3)(OH)-CH_3 + HCl \xrightarrow{ZnCl_2} CH_3-C(CH_3)(Cl)-CH_3 + H_2O$$

2-methylpropan-2-ol → 2-chloro, 2-methyl propane

7 Using the above information, suggest what might have caused the cloudiness in the reactions.

The haloalkanes produced in the reaction are less miscible in water than the alcohols because they do not form hydrogen bonds.

Inv 8.3 Preparation of 2-chloro-2-methylpropane PowerPoint

Haloalkanes can be prepared by nucleophilic substitution of the corresponding alcohol using a suitable halogenating agent

Note: your teacher will show you how to use the equipment safely.

1 Mix 10 mL of methylpropan-2-ol with 20 mL of concentrated hydrochloric acid (corrosive) in a separating funnel. What do you observe?

Two layers are formed and the lower (acid) layer looked cloudy. Gas evolved.

2 Shake for 10 minutes and allow the mixture to settle and leave overnight. What do you observe?

Two distinct layers.

3 Run off the acid layer into a beaker and discard it. Slowly add 10 mL of sodium bicarbonate solution. What was the purpose of this? What was the gas given off?

To neutralise the excess acid.

The gas is carbon dioxide.

4 Observe the mixture. What do you notice?

There are still two layers.

5 Shake gently and release the pressure build-up frequently. Discard the aqueous layer. Run the haloalkane into a test tube, add a spatula tip of anhydrous sodium sulfate. Stopper the tube and shake. What is the purpose of this process?

To absorb any water remaining in the haloalkane layer.

6 Use a data book to find the boiling point of 2-chloro-2-methylpropane.

Boiling point: 51 °C

7 Record the mass of an empty receiving flask ready for distillation.

Mass: 66.7 g

8 Decant the sample into a clean distillation flask and distil it, collecting the fraction that distils within 2 °C of the expected boiling point. Weigh the flask and distillate.

Mass: 73.2 g

9 Complete this table with your experimental data.

	Volume	Density	Mass	Molar mass	Amount present
methylpropan-2-ol	10.0 mL	0.78 g mL^{-1}	7.8 g	74.1 g mol^{-1}	0.105 mol
2-chloro 2-methylpropane			6.5 g	92.6 g mol^{-1}	0.0702 mo

10 Write an equation for the overall reaction.

$$(CH_3)_3COH + HCl \longrightarrow (CH_3)_3CCl + H_2O$$

11 Calculate the expected yield of 2-chloro 2-methylpropane.

$n((CH_3)_3CCl) = n((CH_3)_3COH) = 0.105$ mol

amount = 0.105 mol

12 Calculate the percentage yield of the experiment.

$$\text{Percentage yield} = \frac{\text{actual yield}}{\text{expected yield}} \times 100\%$$

% Yield: 66.9%

$$= \frac{0.0702 \text{ mol}}{0.105 \text{ mol}} \times \frac{100}{1} = 66.9\%$$

Inv 8.4 Hydrolysis of haloalkanes (alkyl halides)

Haloalkanes can readily be converted to alcohols by substitution and to alkenes by elimination.

1 Place 1 mL of ethanol into 3 separate test tubes and place in a beaker of water at about 60 °C.

2 Add 5 drops of 1-chlorobutane, 1-bromobutane and 1-iodobutane (or similar) into the separate test tubes followed by about 2 mL of silver nitrate solution. Shake the test tubes and leave in the 'water bath' and observe over several minutes. What do you observe?

1-chlorobutane: Became a little cloudy after 5 minutes

1-bromobutane: Became cloudy after about 1 minute and after 5 minutes a definite cream precipitate had formed.

1-iodobutane: Rapidly forms a pale yellow precipitate.

3 Using your observations, place the haloalkanes in order of decreasing reactivity.

1-iodobutane > 1-bromobutane > 1-chlorobutane

4 Using the bond energies in the table below, suggest a possible explanation for the different rates of hydrolysis.

Bond	Average bond energy kJ mol^{-1}
C—Cl	339
C—Br	285
C—I	218

As the reactions involve bond breaking the weaker C-I bond is the most easily broken and the C-Cl bond the least easily broken. The experimental results show that the rate of reaction is related to the bond strength – the more easily the bond is broken the faster the reaction rate.

Inv 8.5 Properties of amines PowerPoint

Amines can be considered as derivatives of ammonia with one or more hydrogen atoms replaced by alkyl groups, and they have similar properties. Caution: Amines have strong, lingering smells. Take care not to spill the amines, especially on your school uniform.

1 Cautiously smell the amines and ammonia provided. Describe the smells and compare them.

Ammonia has its own characteristic 'Handy Andy' smell, while the amines smell of a mixture of fish and ammonia.

2 Hold small pieces of damp red and blue litmus above the amines and the ammonia. Note the results.

Both amines and ammonia turn red litmus blue (no change with blue litmus).

3 Place a drop of each amine solution and ammonia on separate pieces of universal indicator paper. Estimate and record the pHs.

Amines and ammonia are both alkaline, but the amines have slightly higher (more purple) pHs than ammonia.

4 Write a general equation for the reaction of an amine with water to explain the pH values.

$R\text{-}NH_2 + H_2O \rightleftharpoons R\text{-}NH_3^+ + OH^-$

5 Prepare separate test tubes containing copper sulfate solution (about 0.5 mL). Add 3 drops of an amine solution or ammonia to each of the test tubes. Mix each solution and describe what you observe.

Ammonia: a pale blue precipitate forms ($Cu(OH)_2$)

Amine: a pale blue precipitate forms ($Cu(OH)_2$)

6 Now add an excess of each amine or ammonia to the appropriate test tube, mix the solutions vigorously, and again record your observations.

Ammonia: the precipitate dissolves and a deep royal blue solution forms.

Amine: the precipitate dissolves and a deep royal blue solution forms.

The amine solution appears darker than the ammonia solution.

7 Write an equation for the reaction between ethylamine and Cu^{2+}(aq).

$Cu^{2+}_{(aq)} + 4C_2H_5NH_{2\,(l)} \longrightarrow [Cu(C_2H_5NH_2)_4]^{2+}_{(aq)}$

Inv 8.6 Reactions of aldehydes and ketones PowerPoint

Aldehydes are better reducing agents than ketones. This property can be used to distinguish between the two classes of compound.

1 Tollens reagent: To 1 mL of silver nitrate solution add 1 drop of sodium hydroxide solution to form a precipitate of silver oxide. Add dilute ammonium hydroxide solution, drop by drop, till the brown precipitate just disappears. Pour this solution into a clean test tube.

Add 3 drops of ethanal (acetaldehyde) and warm the test tube in a beaker of hot water. What do you observe? (Alternatively a black precipitate forms.)

A 'silver mirror' was formed on the inside wall of the test tube.

Write the half-equation for the reduction reaction.

$Ag^+(aq) + e^- \longrightarrow Ag(s)$

2 Fehlings reagent: To 1 ml of solution A (copper sulfate solution) add solution B (an alkaline solution of sodium potassium tartrate) until the blue precipitate just redissolves to give a deep blue solution. (Benedict's reagent can be used instead.)

Add 3 drops of ethanal (acetaldehyde) and boil the mixture for a few minutes. What do you observe?

The blue solution changes to a red-brown precipitate

Write two half-equations, and hence the overall ionic equation for the reaction in alkaline conditions. (The organic product will be a negative ion.)

$CH_3CHO(aq) + 3OH^-(aq) \rightarrow CH_3COO^-(aq) + 2H_2O(l) + 2e^-$

$2Cu^{2+}(aq) + 2OH^-(aq) + 2e^- \rightarrow Cu_2O(s) + H_2O(l)$

$CH_3CHO(aq) + 2Cu^{2+}(aq) + 5OH^-(aq) \rightarrow CH_3COO^-(aq) + Cu_2O(s) + 3H_2O(l)$

3 To 5 drops of potassium dichromate solution (or potassium permanganate solution) add 10 drops of dilute sulphuric acid solution followed by 5 drops of ethanal. Warm carefully on a water bath. What do you observe?

The orange dichromate solution turns green/blue

Write two half-equations, and hence the overall ionic equation for the reaction.

$CH_3CHO(aq) + H_2O(l) \rightarrow CH_3COOH(aq) + 2H^+(aq) + 2e^-$

$Cr_2O_7^{2-}(aq) + 14H^+(aq) + 6e^- \rightarrow 2Cr^{3+}(aq) + 7H_2O(l)$

$3CH_3CHO(aq) + Cr_2O_7^{2-}(aq) + 8H^+(aq) \longrightarrow 3CH_3COOH(aq) + 2Cr^{3+}(aq) + 4H_2O(l)$

4 Repeat the above reactions with using propanone (acetone) in place of ethanal. What do you observe?

No visible change with either oxidising agent.

5 What do the above tests show?

Aldehydes can be oxidised – they act as reducing agents, while ketones are not oxidised.

Inv 9.1 Reactions of acyl chlorides (teacher demo) PowerPoint

Acyl chlorides (acid chlorides) are derivatives of carboxylic acids (alkanoic acids). They are very reactive, undergoing exothermic reactions with water, alcohols and ammonia to give the corresponding carboxylic acids, esters and amides. (These reactions should be done in a fume cupboard or under a fume hood.) All acyl chlorides are corrosive.

1 In a dry test tube place 5 drops of ethanoyl chloride (acetyl chloride). Carefully add 5 drops of water. What do you observe? Test any gas given off with damp litmus.

A vigorous reaction with gas given off. The reaction mixture gets very hot. The damp litmus turns red.

2 Add a few drops of silver nitrate solution. What do you observe? What does this indicate?

A white precipitate forms, indicating the presence of Cl^- ions.

3 Write an equation for the reaction between ethanoyl chloride and water

$CH_3COCl(l) + H_2O(l) \longrightarrow CH_3COOH(aq) + HCl(g)$

4 In a dry test tube place 5 drops of ethanoyl chloride. Carefully add 5 drops of ethanol. Shake carefully for about a minute and then carefully add a few drops of water. Carefully smell the product. Describe the smell.

A distinctive smell similar to glue.

5 Write an equation for the reaction between ethanoyl chloride and ethanol. Name the products.

$CH_3COCl(l) + CH_3CH_2OH(l) \longrightarrow CH_3COOCH_2CH_3 + HCl$

ethyl ethanoate hydrogen chloride

6 (IN A FUME CUPBOARD) Put 10 mL of 0.880 ammonia in a 100 mL beaker, then carefully add a few drops of ethanoyl chloride. What do you observe?

A vigorous reaction and a lot of white fumes.

7 What are the fumes that are given off? Ammonium chloride

8 Write a balanced equation for the reaction between ethanoyl chloride and ammonia and name the products.

ethanamide hydrogen chloride

$CH_3COCl(l) + NH_3(aq) \longrightarrow CH_3CONH_2 + HCl$

or $CH_3CONH_3^+Cl^-(s)$

Inv 9.2 Amide hydrolysis

Amides can be hydrolysed either by acid or alkali.

1 In a test tube place a few crystals of ethanamide. Add 3 to 4 mL of dilute sodium hydroxide solution. Warm gently and then carefully smell the gas produced. Observation:

The gas smells of ammonia.

2 Test the gas with damp red and blue litmus. Observation:

The litmus turns blue.

3 Write an equation for the reaction.

$CH_3CONH_2(s) + NaOH(aq) \longrightarrow CH_3COO^-Na^+(aq) + NH_3(g)$

4 Cool and keep the mixture.

5 Now add 4 mL, a slight excess, of dilute sulfuric acid and warm the test tube and smell carefully. Observation:

It smells of ethanoic acid (vinegar).

6 What gas is formed? Ethanoic acid (CH_3COOH)

7 What effect does it have on litmus? It turns litmus red.

8 Write an equation for the reaction.

$CH_3COO^-(aq) + H^+(aq) \longrightarrow CH_3COOH(aq)$ (Some comes off as gas)

9 Take a fresh sample of ethanamide. Add 2 mL of dilute sulfuric acid. Warm gently and then carefully smell the gas produced. Observation:

It smells of ethanoic acid.

10 Test the gas with damp red and blue litmus. Observation:

The blue litmus turns red.

11 Write an equation for the reaction.

$CH_3CONH_2(s) + H_3O^+(aq) \longrightarrow CH_3COOH(aq) + NH_4^+(aq)$

12 Cool and keep the mixture.

13 Now add 4 mL of sodium hydroxide solution. Warm the test tube and smell carefully. Observation:

There is a smell of ammonia.

14 What gas is formed? Ammonia (NH_3)

15 What effect does it have on litmus? It turns litmus blue.

16 Write an equation for the reaction.

$NH_4^+(aq) + OH^-(aq) \longrightarrow NH_3(g) + H_2O(l)$

Inv 9.3 Identification of organic unknowns

Classes of organic compounds have characteristic physical and chemical properties due to the functional group(s) they contain. Unknown compounds may be identified using these characteristics.

1 Give the physical or chemical characteristics of the following classes of compounds that will allow you to distinguish between them.

Characteristics of alkanes

Insoluble in water; less dense than water; do not conduct electricity; will burn; neutral; reacts slowly with bromine in light, no reaction with $KMnO_4$.

Characteristics of alkenes

As for alkanes except – burns with smokier flame, rapidly decolorises bromine water in dark, decolorises H^+/MnO_4^-.

Characteristics of alkyl halides

Insoluble in water, alkaline hydrolysis to give $X^-(aq)$ (test with silver nitrate solution).

Characteristics of alcohols

Low-mass soluble in water, neutral, clean flame, 1° + 2° oxidised by H^+/MnO_4^- or $H^+/Cr_2O_7^{2-}$, react with R-COOH to form esters, 3°, 2° react with Lucas reagent. not oxidised by Tollens or Fehling's solutions.

Characteristics of aldehydes

Low-mass soluble in water, oxidised by H^+/MnO_4^-, $H^+/Cr_2O_7^{2-}$, Tollens + Fehlings.

Characteristics of ketones

Low-mass soluble in water Not oxidised by oxidising agents.

Characteristics of amines

Typical basic properties – blue to litmus, form salts with acid, smells of ammonia + fish. forms royal blue complex with Cu^{2+}.

Characteristics of carboxylic acids

Typical acidic properties (metals, carbonates, litmus) miscible in water, reacts with alcohols to form esters. strong smells.

Characteristics of esters

Not miscible in water, characteristic fruity smells.

Characteristics of acid chlorides

Fume in moist air, react with water to produce $HCl(g)$ (test with litmus), rapidly form esters with alcohols.

Characteristics of amides

Low melting-point solids, mousey smell, react with OH^- to produce NH_3 (test with litmus) or with H_3O^+ to produce carboxylic acids (smell + test with litmus).

2 Use the characteristics you have listed to identify each of the 4 unknown compounds your teacher will give you, labelled W, X, Y and Z. Present your results in the form of a flow-diagram or table that includes all relevant information. Write equations for any chemical reactions that occur.

1) Hold damp red litmus above each liquid.	Y turns litmus blue. No change with others.	Y probably amine.

$$CH_3CH_2NH_2(l) + H_2O(l) \longrightarrow CH_3CH_2NH_3^+(aq) + OH^-(aq)$$

2) Add 1 mL of Y to 2 mL of copper sulfate solution.	A pale blue ppt forms the dissolves in a royal blue complex	Y = aminoethane

$$4CH_3CH_2NH_2(l) + Cu^{2+}(aq) \longrightarrow [Cu(CH_3CH_2NH_2)_4]^{2+}(aq)$$

3) Add 1 mL of remaining liquids each to 2 mL of sodium carbonate solution.	W dissolves 1 layer. X 2 layers Z fizzes	W = alcohol, ketone or aldehyde. X = methyl ethanoate Z = propanoic acid.

$$CH_3CH_2COOH(l) + H_2O(l) \rightleftharpoons CH_3CH_2COO^-(aq) + H_3O^+(aq)$$

$$2H_3O^+(aq) + CO_3^{2-}(aq) \longrightarrow CO_2(g) + 3H_2O(l)$$

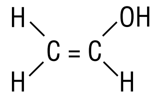
4) Warm 1 mL W with acidified dichromate.	Orange soln turns blue-green.	W = alcohol or aldehyde.
5) Warm 1 mL W with Tollens reagent.	Silver mirror	W = aldehyde W = methanal.

$$HCHO \xrightarrow{H^+/Cr_2O_7^{2-}} HCOOH$$

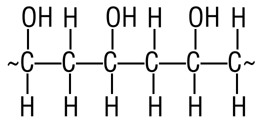
$$HCHO \xrightarrow{[Ag(NH_3)_2]^+} HCOOH$$

Inv 9.4 Making an addition polymer: slime

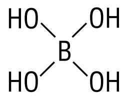
Vinyl alcohol (or 1-hydroxyethene) undergoes addition polymerisation to form the linear polymer polyvinyl alcohol. Slime is made from polyvinyl alcohol and borax, which contains the borate ion. The polar –OH group on the polymer and the –OH group on the borate ion undergo hydrogen bonding. The linear chains are thus held together in a loose network with water trapped between them. The result is a gel-like slime in which the hydrogen bonds can break and reform easily.



vinyl alcohol



polyvinyl alcohol



borax

1 Pour 50 mL of 6% polyvinyl alcohol into a 100 mL beaker and add a few drops of food colouring.

2 Add 10 mL of 4% borax solution and stir with a glass rod. (It takes a few minutes for the slime to appear.)

3 Describe the properties of the slime.

It is a transparent, thick gel with the colour of the food colouring.
It is quite squishy like very pliable rubber or soft bubblegum

4 How can the properties of the slime be explained by its structure and bonding?

The polymer molecules form long chains which tangle together as they make and break hydrogen bonds.

Inv 9.5 Making a condensation polymer: nylon 6, 6 PowerPoint

Nylon 6, 6 is one type of copolymer formed by the condensation polymerisation of two monomers: hexanedioyl (adipyl) chloride and 1,6-diaminohexane, hydrogen chloride being eliminated.

1 Pour 10 mL of adipyl chloride (corrosive) (5% solution in hexane) into a beaker that has been coated on the inside with paraffin oil. (Dye can be added if desired.)

2 Add 5 g of hydrated sodium carbonate and stir with a glass rod.

3 What is the purpose of the sodium carbonate?

It will neutralise the HCl produced during the condensation reaction.

3 Carefully add 10 mL of the 5% solution of 1,6-diaminohexane so that the two solutions do not mix.

A small amount of solid will form at the interface of the two liquids.

4 Using a glass rod or tweezers, pull out a little of this solid and carefully wind the thread around a glass rod. (The thread will go on forming until the solutions are used up.)

5 Write an equation using structural formulae to illustrate this condensation reaction.

$H_2N-(CH_2)_6-NH_2 + ClOC(CH_2)_4COCl \longrightarrow \sim[NH(CH_2)_6NH-CO(CH_2)_4CO]\sim + 2HCl$

1,6 diaminohexane — adipyl chloride — nylon 6,6 fragment

Inv 10.1 Determination of the solubility of barium hyroxide

The solubility of barium hydroxide can be determined by titration of a saturated solution with standard sulfuric acid solution.

Note: barium hydroxide is toxic, and the solid is also corrosive. Do not touch the solid, and wash your hands after the experiment.

1 Prepare a saturated solution of barium hydroxide by adding about 5 g of $Ba(OH)_2.8H_2O$ to 60 mL of water and shaking periodically for about 10 minutes. Filter the solution and pipette 10 mL aliquots of the filtrate into conical flasks.

2 Titrate with standard H_2SO_4 using methyl orange indicator.

Burette reading	1st titration	2nd titration	3rd titration	4th titration
Initial reading	2.2 mL	10.5 mL	18.4 mL	26.2 mL
Final reading	10.3 mL	18.4 mL	26.1 mL	34.0 mL
Titre	~~8.1 mL~~	7.9 mL	7.7 mL	7.8 mL

3 Calculate the average of concordant titres. $V(H_2SO_4)$ = 7.8×10^{-3} L

4 What was the concentration of the sulfuric acid used? $c(H_2SO_4)$ = 0.200 mol L^{-1}

5 Calculate the amount, in moles, of acid used. $n = cV = 0.200 \times 7.8 \times 10^{-3} = 1.56 \times 10^{-3}$ mol $n(H_2SO_4)$ = 1.56×10^{-3} mol

6 Write a balanced equation for the reaction between barium hydroxide and sulfuric acid.

$Ba(OH)_2(aq) + H_2SO_4(aq) \longrightarrow BaSO_4(s) + 2H_2O(l)$

7 Complete the expression:

$$\frac{n(Ba(OH)_2)}{n(H_2SO_4)} = \frac{1}{1}$$

$$n(Ba(OH)_2) = 1 \times n(H_2SO_4)$$

8 Calculate the amount, in moles, of $Ba(OH)_2$ present. $n(Ba(OH)_2)$ = 1.56×10^{-3} mol

9 Calculate the concentration of the $Ba(OH)_2$)solution. $c = \frac{n}{V} = \frac{1.56 \times 10^{-3}}{10 \times 10^{-3}} = 0.156$ mol L^{-1} $c(Ba(OH)_2)$ = 0.156 mol L^{-1}

10 If the molar mass of the barium hydroxide is 315.5 g mol^{-1} calculate the solubility in g L^{-1}.

S = 0.156 mol L^{-1} × 315.5 g mol^{-1}

= 49.2 g L^{-1}

11 How does this value compare with the data table value? Comment on any difference.

The book value is 4.7g/100mL, which is 47 g L^{-1}. The difference may be due to the solubility at different temperatures or to some undissolved material passing through the filter paper.

Inv 10.2 Solubility and solubility products

Silver halides are sparingly soluble in water and silver ions form an ammine complex with ammonia.

1 Place 2 mL of silver nitrate solution into each of 3 test tubes.

2 To the first tube add 3 or 4 drops of potassium chloride solution, to the second add the same amount of potassium bromide solution and to the third add the same amount of potassium iodide solution. What do you observe in each case? Write balanced ionic equations for any reactions.

Chloride: A white precipitate formed.

$Ag^+(aq) + Cl^-(aq) \rightleftharpoons AgCl(s)$

Bromide: A cream precipitate formed.

$Ag^+(aq) + Br^-(aq) \rightleftharpoons AgBr(s)$

Iodide: A pale yellow precipitate formed.

$Ag^+(aq) + I^-(aq) \rightleftharpoons AgI(s)$

3 Divide the contents of each of the 3 test tubes from **2** into 2 equal parts.

4 To the first sample of each add dilute ammonia solution in excess and shake. What do you observe in each case? Write balanced ionic equations for any reactions.

Chloride: The white precipitate redissolves.

$AgCl(s) + 2NH_3(aq) \rightleftharpoons [Ag(NH_3)_2]^+(aq) + Cl^-(aq)$

Bromide: no visible reaction - precipitate remains

Iodide: no visible reaction - precipitate remains.

5 To the second sample of each add concentrated ammonia solution (corrosive) in excess and shake. What do you observe in each case? Write balanced ionic equations for any reactions.

Chloride: The white precipitate disappears

$AgCl(s) + 2NH_3(aq) \rightleftharpoons [Ag(NH_3)_2]^+(aq) + Cl^-(aq)$

Bromide: The cream precipitate disappears

$AgBr(s) + 2NH_3(aq) \rightleftharpoons [Ag(NH_3)_2]^+(aq) + Br^-(aq)$

Iodide: no visible reaction - precipitate remains

The solubility products of the silver halides are: $K_s(AgCl) = 2 \times 10^{-10}$, $K_s(AgBr) = 5 \times 10^{-13}$, $K_s(AgI) = 8 \times 10^{-17}$.

6 Use the above data to explain your observations.

AgCl, with the greatest K_s, is the most soluble. When the complex forms $[Ag^+]$ decreases to bring IP below K_s so the AgCl dissolves. AgBr is less soluble, but with the greater $[NH_3]$ in conc ammonia increases the amount of complex formed, reducing $[Ag^+]$ until IP < K_s and the AgBr dissolves. AgI is so insoluble that even conc ammonia does not reduce $[Ag^+]$ enough to dissolve AgI.

Inv 10.3 The common ion effect PowerPoint

If the ionic product is greater than the solubility product then precipitation can occur. The introduction of a common ion upsets the equilibrium.

1 Pour about 5 mL of saturated salt solution into a clean test tube.
How do you know that the solution is saturated?

Because there is undissolved salt in the bottom of the bottle of saturated salt solution.

2 Add concentrated hydrochloric acid (corrosive) drop by drop. What do you notice?

A white crystalline solid immediately forms.

3 Write the equilibrium equation for the saturated sodium chloride:

$NaCl_{(s)} \rightleftharpoons Na^+_{(aq)} + Cl^-_{(aq)}$

4 Write the expression for the equilibrium constant, K_s:

$K_s = [Na^+][Cl^-]$

5 List the ions present in concentrated hydrochloric acid solution $H_3O^+_{(aq)}$ $Cl^-_{(aq)}$ $OH^-_{(aq)}$

6 Name the common ion that has been added: $Cl^-_{(aq)}$

7 What does this do to the ionic product?

It increases the ionic product, making IP > Ks so a precipitate forms

8 What can you say about the solubility of a compound in the presence of another compound with a common ion?

The compound is less soluble in the presence of another compound with a common ion.

Inv 11.1 Species in solution

An acid or base can be an ion or a neutral molecule.

1 To each of the following approximately equal concentration solutions add 2 drops of universal indicator. Record your observations in the table below.

Solution	Colour in Universal Indicator	pH
Ammonium chloride NH_4Cl	orange	5
Sodium hydrogen carbonate $NaHCO_3$	blue-green	9
Sodium dihydrogen phosphate NaH_2PO_4	yellow	6
Sodium carbonate Na_2CO_3	purple	11
Sodium sulfite Na_2SO_3	blue-green	9

2 Which ions behave as acids towards water? $NH_4^+(aq)$ and $H_2PO_4^-(aq)$

3 Which ions behave as bases towards water? $HCO_3^-(aq)$ $CO_3^{2-}(aq)$ and $SO_3^{2-}(aq)$

4 Which ion is the **strongest acid**? $NH_4^+(aq)$

5 Which ion is the **strongest base**? $CO_3^{2-}(aq)$

6 Write balanced ionic equations to represent the proton transfer reactions that occur when:

a $NaHCO_3$ dissolves in water

$HCO_3^-(aq) + H_2O(l) \rightleftharpoons H_2CO_3(aq) + OH^-(aq)$

b NaH_2PO_4 dissolves in water

$H_2PO_4^-(aq) + H_2O(l) \rightleftharpoons HPO_4^{2-} + H_3O^+(aq)$

c Na_2CO_3 dissolves in water

$CO_3^{2-}(aq) + H_2O(l) \rightleftharpoons HCO_3^-(aq) + OH^-(aq)$

d Na_2SO_3 dissolves in water

$SO_3^{2-}(aq) + H_2O(l) \rightleftharpoons HSO_3^-(aq) + OH^-(aq)$

Inv 11.2 Determination of K_a and K_b

If the total concentration of a weak acid is known, then a determination of the $[H_3O^+]$ at equilibrium will give the concentrations of all species in the equilibrium expression. A numerical value of K_a can then be calculated. Similarly K_b can be determined for a base.

1 Standardise the pH meter (or probe) with a suitable acid buffer (potassium hydrogen tetroxalate – pH 2.16).

2 Rinse the electrode thoroughly in distilled water then measure the pH for the following in the order given. Thoroughly rinse the electrode between each measurement.

Solution	pH	$[H_3O^+]$	K_a (see **3** below)	α (see **5** below)
0.01 mol L^{-1} HCOOH	3.0	1×10^{-3}	$\frac{(1\times10^{-3})^2}{0.01} = 1\times10^{-4}$	$\frac{1\times10^{-3}}{0.01} = 0.1$
0.1 mol L^{-1} HCOOH	2.5	3.16×10^{-3}	$\frac{(3.16\times10^{-3})^2}{0.1} = 9.9\times10^{-5}$	$\frac{3.16\times10^{-2}}{0.1} = 0.03$
1 mol L^{-1} HCOOH	2.0	1×10^{-2}	$\frac{(1\times10^{-2})^2}{1} = 1\times10^{-4}$	$\frac{1\times10^{-2}}{1} = 0.01$

3 From the pH values measured for each of the 3 different HCOOH solutions, calculate the equilibrium $[H_3O^+]$ and hence values for K_a (methanoic or formic acid) using the following:

$$HA + H_2O \rightleftharpoons A^- + H_3O^+$$

$$K_a = \frac{[A^-][H_3O^+]}{[HA]}$$

Assumption: at equilibrium $[H_3O^+] = [A^-]$

4 Compare the 3 values for K_a obtained and comment on how well they agree.

The 3 values are the same within the accuracy of the pH measurements.

5 From the pH values measured for HCOOH, calculate for each of the three solutions the degree of ionisation, $\alpha = [H_3O^+]/y$ (where y = total acid concentration). Comment on the trend in values of α.

The more concentrated the solution the lower the degree of ionisation. (Diluting an acid increases the degree of ionisation.

6 Standardise the pH meter with a suitable basic buffer (borax – pH 9.18).

7 Rinse the electrode thoroughly in distilled water then measure the pH of 1 mol L^{-1} NH_3 solution.

pH = 9.4

8 Calculate $[OH^-]$. $[OH^-]$ = 2.5×10^{-5} mol L^{-1}

$[H_3O^+] = 10^{-pH}$
$= 10^{-9.4}$
$= 3.98 \times 10^{-10}$ mol L^{-1}

$[OH^-] = \frac{1\times10^{-14}}{[H_3O^+]}$
$= \frac{1\times10^{-14}}{3.98\times10^{-10}}$
$= 2.51 \times 10^{-5}$ mol L^{-1}

9 Use the information below to calculate a value for K_b.

$$B + H_2O \rightleftharpoons BH^+ + OH^-$$

$$K_b = \frac{[BH^+][OH^-]}{[B]}$$

Assumption: at equilibrium $[BH^+] = [OH^-]$

$NH_3(aq) + H_2O(l) \rightleftharpoons NH_4^+(aq) + OH^-(aq)$

$K_b = \frac{[NH_4^+][OH^-]}{[NH_3]}$

Assume $[NH_4^+] = [OH^-]$
$[NH_3] = 1\ mol\,L^{-1}$

$K_b = \frac{[OH^-]^2}{1}$

$= \frac{(2.5 \times 10^{-5})^2}{1}$

$= 6.3 \times 10^{-10}$

$K_b =$ 6.3×10^{-10}

Inv 12.1 Buffer action

This experiment demonstrates the buffer action (or lack of it) for selected systems when small amounts of a strong acid or strong base are added to these systems and the change in pH noted.

1 Measure the pH of aliquots (measuring cylinder) of the following solutions and note the change in pH after addition by pipette of 1 mL of strong acid and/or strong base (2 mL of NaOH for the last two) provided.

Solution	pH	pH after adding 1 mL of 0.2 mol L^{-1} HCl	pH after adding 1 mL of 0.2 mol L^{-1} NaOH
40 mL distilled water	7.0	2.3	
40 mL of 10^{-3} mol L^{-1} HCl	3.0		11.6
100 mL of sea water $[HCO_3^-] = 0.004$ mol L^{-1} and $[CO_3^{2-}] = 0.006$ mol L^{-1}	Seawater 8.3 Carbonate 9.4		Seawater 8.6 Carbonate 9.8
40 mL of 0.1 mol L^{-1} ethanoic acid /0.1 mol L^{-1} sodium ethanoate solution	4.8	4.6	5.0
40 mL of 0.1 mol L^{-1} ammonium chloride / 0.1 mol L^{-1} ammonia solution	9.5	9.3	9.7

2 How does the behaviour of buffer solutions differ from that of other solutions?

In water and in dilute acid addition of 1 mL of acid or base produces a large change in pH, but in each of the buffer mixtures addition of the same amount of acid or base produces only small changes in pH of only ± 0.2 - 0.4 pH units.

3 Write a brief explanation for their buffer action.

Buffers contain a weak acid and its conjugate base, or a weak base and its conjugate acid. They "mop up" excess H_3O^+ or OH^- by converting acid to base or base to acid.

eg $NH_3(aq) + H_3O^+(aq) \rightleftharpoons NH_4^+(aq) + H_2O(l)$

$NH_4^+(aq) + OH^-(aq) \rightleftharpoons NH_3(aq) + H_2O(l)$

4 Milk contains, in addition to fats and proteins, a mixture of lactic acid ($CH_3CH(OH)COOH$), calcium and potassium lactates, amino acids, $H_2PO_4^-$ and HPO_4^{2-}. Will it show buffer action? Explain your answer.

Yes, because it contains lactic acid (HLac) and lactates (Lac^-).

$HLac + OH^- \rightleftharpoons Lac^- + H_2O$

$Lac^- + H_3O^+ \rightleftharpoons HLac + H_2O$

Inv 12.2 Titration curves PowerPoint

During an acid-base titration the pH changes in a characteristic way. A titration curve is a graph which shows the change in pH as the titration proceeds.

1 Fill a burette with sodium hydroxide solution (0.1 mol L^{-1}).

2 Pipette 20 mL of hydrochloric acid (or ethanoic acid), 0.1 mol L^{-1}, into a 100 mL beaker. Place a magnetic stirrer in the solution and place on a hotplate/stirrer.

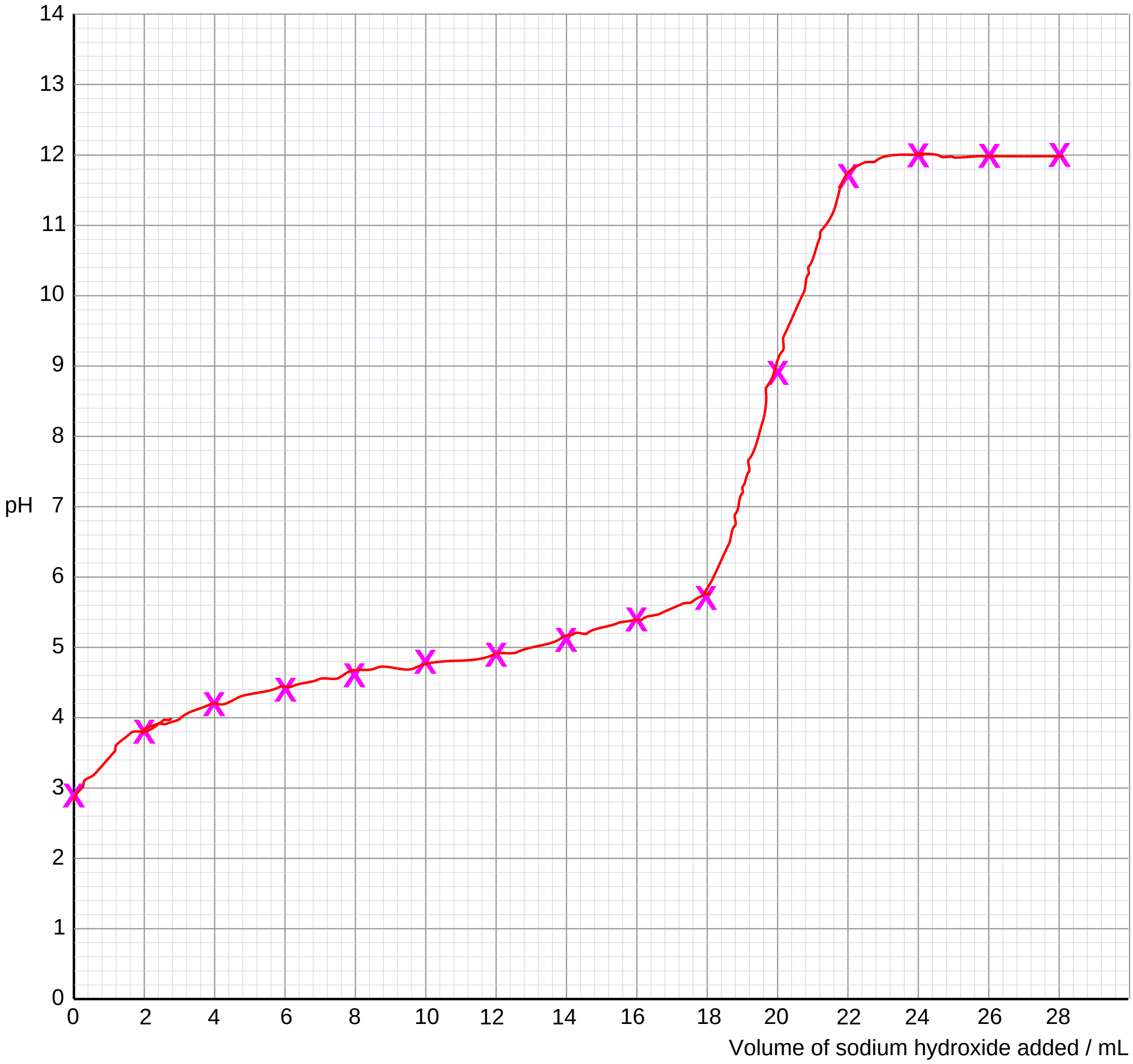
3 Use a pH meter or pH data logger probe to measure the pH at the start. pH = 2.9

4 Add 2.0 mL of the sodium hydroxide solution and measure the pH. pH = 3.8

5 Continue to add the sodium hydroxide solution 2. 0 mL at a time and record the pH each time.

Volume of NaOH (mL)	pH	Volume of NaOH (mL)	pH	Volume of NaOH (mL)	pH
0	2.9	10	4.8	20	8.9
2	3.8	12	4.9	22	11.7
4	4.2	14	5.1	24	12.0
6	4.4	16	5.4	26	12.0
8	4.6	18	5.7	28	12.0

6 On the grid below plot a graph of pH (vertical axis) against volume of sodium hydroxide added. Join the points with a smooth curve of best fit.



7 What is the initial pH of the acid? pH = 2.9

8 How many mL of sodium hydroxide were added at the equivalence point? V(NaOH) = 20.0 mL

(7.0 for HCl)

9 What is the pH at the equivalence point? pH = 9.0

10 Name a suitable indicator for the reaction. phenolphthalein
(bromothymol blue for HCl titration)

Inv 13.1 A back titration

Many reactions are not suitable for analysis by a standard titration, perhaps because the reaction is very slow, or because other reaction products form that get in the way of an exact result. Chemists often avoid these problems by doing a *back titration.*

Calcium carbonate is a common ingredient in indigestion tablets. It reacts with acid, but the reaction with solid $CaCO_3$ takes time, and the CO_2 produced is acidic.

These factors make a direct titration difficult. Instead, we react the crushed tablets with a known volume of excess of standard HCl, heat the mixture to remove all CO_2, and then titrate in standard NaOH until the rest of the acid has reacted.

1 Crush a Quick-Eze tablet and transfer completely to a 100 mL conical flask. Repeat for two other tablets.

2 Pipette 20.0 mL (excess) of standard hydrochloric acid into each flask.

3 Boil the mixtures for 2 minutes to remove all the CO_2, and allow to cool..

4 Write an equation for the reaction between HCl and $CaCO_3$.

$CaCO_3(s) + 2HCl(aq) \rightarrow CaCl_2(aq) + H_2O(l) + CO_2(g)$

5 Write an equation for the reaction between HCl and NaOH.

$NaOH(aq) + HCl(aq) \rightarrow NaCl(aq) + H_2O(l)$

6 Titrate the excess hydrochloric acid against standard sodium hydroxide from the burette using phenolphthalein indicator.

Burette reading	**1st tablet**	**2nd tablet**	**3rd tablet**
Initial reading	1.3 mL	19.7 mL	5.4 mL
Final reading	19.7 mL	37.9 mL	24.0 mL
Titre	18.4 mL	18.2 mL	18.6 mL

			1st tablet	2nd tablet	3rd tablet
7	Record the concentration of hydrochloric acid added.	c(HCl) =	1.073 mol L^{-1}	1.073 mol L^{-1}	1.073 mol L^{-1}
8	Record the volume of hydrochloric acid added.	V_{Tot}(HCl) =	20.0×10^{-3} L	20.0×10^{-3} L	20.0×10^{-3} L
9	Calculate the amount of hydrochloric acid added.	n_{Tot}(HCl) =	2.0146×10^{-2} mol	2.0146×10^{-2} mol	2.0146×10^{-2} mol
10	Record the concentration of NaOH used.	c(NaOH)	0.484 mol L^{-1}	0.484 mol L^{-1}	0.484 mol L^{-1}
11	Record the volume of NaOH required.	V(NaOH) =	18.4×10^{-3} L	18.2×10^{-3} L	18.6×10^{-3} L
12	Calculate the amount of NaOH required for each tablet.	n(NaOH) =	8.906×10^{-3} mol	8.809×10^{-3} mol	9.002×10^{-3} mol

13	Use the titration data to calculate the amount of HCl reacting with the NaOH.	$n_{unreacted}(HCl)$ = $n(NaOH)$ = (from 10)	8.906×10^{-3} mol	8.809×10^{-3} mol	9.002×10^{-3} mol
14	Calculate the amount of HCl reacting with the $CaCO_3$ in the tablets.	$n(HCl)$ = $n_{TOT}(HCl) - n_{unreacted}(HCl)$ box 7-10	1.124×10^{-2} mol	1.134×10^{-2} mol	1.114×10^{-2} mol
15	Calculate the amount of $CaCO_3$ present in each tablet.	$n(CaCO_3)$ = $\frac{1}{2} n(HCl)$ =	5.62×10^{-3} mol	5.67×10^{-3} mol	5.57×10^{-3} mol
16	Calculate the mass of calcium carbonate in each tablet. $M(CaCO_3)$ = 100.0 g mol^{-1}.	$m(CaCO_3)$ = nM =	0.562 g	0.567 g	0.557 g

17 Calculate the average mass of calcium carbonate in the Quick-Eze tablets.

$m_{av}(CaCO_3) = \frac{0.562\,g + 0.567\,g + 0.557\,g}{3} = 0.562\,g$

Inv 13.2 Colorimetric analysis

Brass is an alloy of copper and zinc. It dissolves in nitric acid. The amount of copper in a sample can be found by colorimetry.

1 Weigh accurately about 1 g of brass. m(brass) = 1.008 g

2 Dissolve the metal in concentrated nitric acid (corrosive) using the minimum amount of acid possible. WORK IN A FUME CUPBOARD.

3 Transfer the solution to a 100.0 mL volumetric flask and make it up to volume with dilute sulfuric acid.

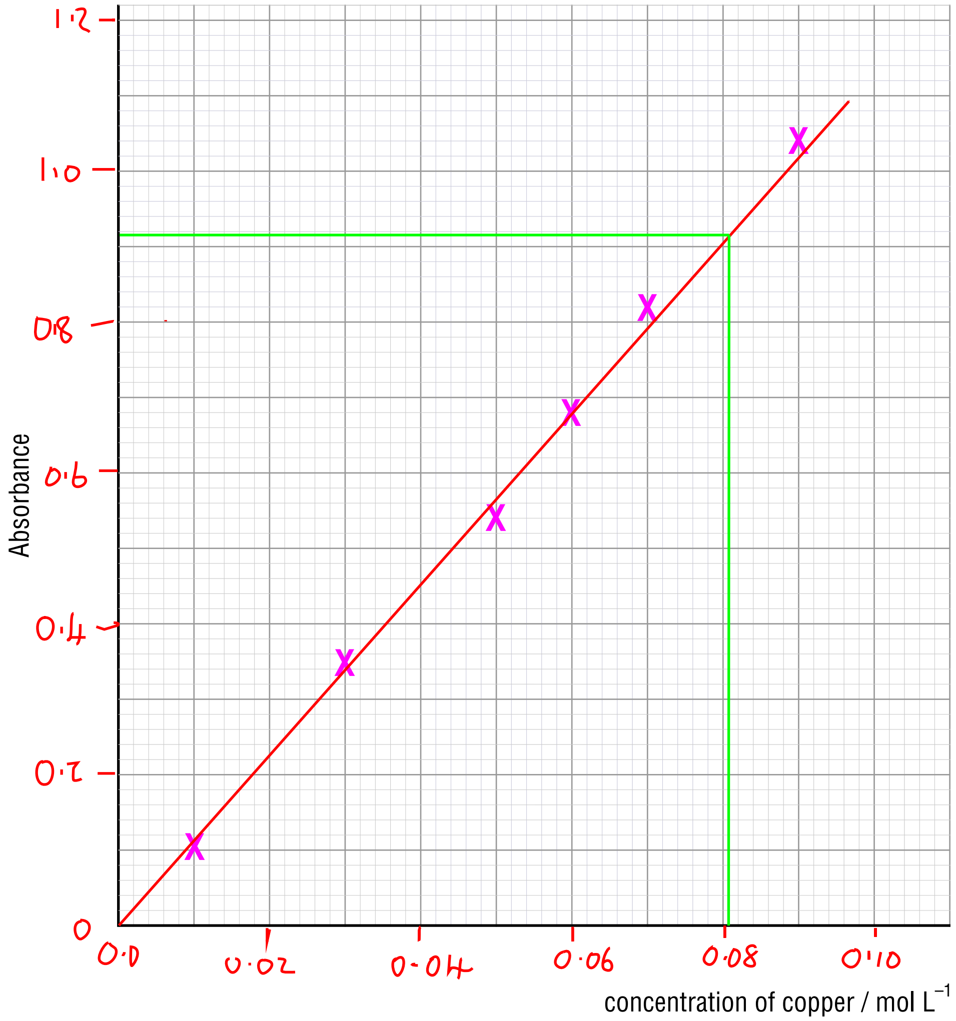
4 Use a standard 0.100 mol L^{-1} copper sulfate solution to prepare standard copper ion solutions as shown below

Concentration (mol L^{-1})	0.010	0.030	0.050	0.060	0.070	0.090
Volume of 0.100 mol L^{-1}) solution required	1 mL	3 mL	5 mL	6 mL	7 mL	9 mL
Volume of dilute sulfuric acid required	9 mL	7 mL	5 mL	4 mL	3 mL	1 mL

5 Measure the absorbance of each standard solution, and the sample, using a colorimeter with a red filter. Set the colorimeter to 0 absorbance, or take the base measurement, for the dilute sulfuric acid.

Solution (mol L^{-1})	0.010	0.030	0.050	0.060	0.070	0.090	Sample
Absorbance	0.112	0.348	0.540	0.680	0.820	1.040	0.918

6 Plot a calibration curve of your results and use it to determine the concentration of copper ions in the sample solution



$c(Cu^{2+})$ = 0·083 mol L^{-1}

7 Calculate the amount, in moles, of copper in the 100.0 mL flask.

$n = cV$

$n(Cu^{2+}) = 0{\cdot}083 \text{ mol L}^{-1} \times 100{\cdot}0 \times 10^{-3} \text{ L}$

$= 0{\cdot}0083 \text{ mol}$

8 Calculate the mass of copper in the brass sample.

$m = nM$

$m(Cu) = 0{\cdot}0083 \text{ mol} \times 63{\cdot}5 \text{ g mol}^{-1}$

$= 0{\cdot}527 \text{ g}$

9 Calculate the percentage of copper in the brass.

$\% Cu = \frac{m(Cu)}{m(brass)}$

$= \frac{0{\cdot}527 \text{ g}}{1{\cdot}008 \text{ g}} \times \frac{100}{1} = 52\%$