CHEMISTRY LEVEL 4C
(CHM415115)

LABORATORY MANUAL

EXPERIMENTAL PROCEDURES

&

REVIEW   QUESTIONS

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INDEX:

CHEMISTRY (LEVEL 4C)
LABORATORY REPORTS

INTRODUCTION:

The laboratory exercises associated with this subject constitute a vital section of your knowledge development in the sciences. Laboratory work is not only an essential part of this course through enhancing your understanding of the scientific principles involved, it is also a key component of the assessment process.

Laboratory reports are important and need to be submitted for marking whenever your teacher requests them. You MUST have a completely separate exercise **book** or **folder** for your lab. reports. Don't use pad paper please.

We recommend that at the front of your laboratory note book you keep an up to date index of the experiments that you have performed and recorded.

TYPES OF WRITTEN REPORTS:

During the year, there are likely to be three different situations arising with regard to report writing. These are:

- reports completed in class where a **limited** discussion is required and the teacher provides guidance for this.

- reports completed outside normal class time where a **detailed** discussion of results is expected from you.

- reports **completed in class** where a detailed discussion is required from you and this is written under 'test' conditions.

NOTE ON CRITERION 2:

"Develop and Evaluate Experiments"

This criterion is one where your laboratory reports will play an important role in the assessment process. This will become clearer when you consider the suggestions for the format and headings in your report writing, given on the next page.

REPORT FORMAT:

Although there is not necessarily any one 'correct' method for writing laboratory reports in Chemistry, the preferred format is one involving the following headings:

1. **HEADER**: This should include the name(title) of the experiment and the date(s) on which the experiment was performed. All experiments should be commenced on a new page of your laboratory report book.

2. **AIM**: This should indicate to the reader, the intent of the experiment; e.g. "To determine the acceleration due to gravity by observing the motion of an object moving down an inclined plane."

3. **PROCEDURE**: This should explain to the reader how the experiment was carried out. In most instances, the procedure will come directly from this manual. You do NOT need to rewrite this procedure section. In some cases however, you may need to give a reference to the text from which the procedure was taken. e.g. refer to: Gould, M.E. CHEMISTRY LABORATORY MANUAL, McGraw-Hill, 1996, pp 126 -128.

4. **OBSERVATIONS:** This section lists results and all the information and data you gathered. Observations in the laboratory involve seeing, hearing, touching, smelling **but not tasting**!

5. If possible, show the collected information in the form of a table.
Tabulation is particularly desirable where numerical data are to be recorded. In this case, rule up the table and label the columns showing the quantity and units where appropriate. Units only need be shown at the top of the respective data columns.

6. **DISCUSSION OF RESULTS:** This is where the key to the report lies. It normally involves some or all of the following:
   - plotting a graph to show the collected data
   - analysis of the graphical data (finding a mathematical relationship?)
   - calculations based upon the data collected
   - offering explanations(hypotheses) for the results obtained
   - answering given questions relating to the experiment
   - calculating a % error if an accepted numerical answer is achievable
   - discussing the sources of error and their relative significance
   - assumptions that were made in obtaining the hypotheses or results
   - suggestions for possible improvements to the experimental procedure
   - alternate experiments that might be performed to test the validity of your hypothesis(es).

7. **CONCLUSION**: This comes at the very end of your report and offers in 2 or 3 lines only, a *summary* of your findings. Look back to the "aim" of the experiment and this will most likely give you a clue to stating your conclusion. Was the experiment a success or otherwise?
e.g. "The percentage by mass of iron(II)sulfate in the sample was found to be 78.2%. This result was generally in good agreement with the theoretical value as indicated by the incurred error of only 3.6%."

CHEMISTRY (LEVEL 4C)
EXPERIMENT 1.
INTRODUCTION TO "REDOX"

Aim:

To carry out 5 separate experiments that involve electron transfer reactions and then to establish the oxidation and reduction half-equations involved in each. For each redox reaction, we will then write a net-ionic redox equation, a total redox equation and establish the ions that are "spectators".

Procedure Mini-Experiment 1

Place one small granule (~1g) of zinc metal in a test-tube and cautiously add about 10 mL of 5 mol/L hydrochloric acid. Observe and test for any gases that may be evolved.

(i) Write out the total equation for the reaction.

(ii) From this total equation, deduce the two redox half-equations.

(iii) Give the net ionic equation.

(iv) Which is the oxidation 1/2-equation and which is the reduction 1/2-equation?

(v) What are the spectator ions in this reaction?

Procedure Mini-Experiment 2

Place about 5 mL of a 0.1 mol L^{-1} solution of potassium iodide ($KI_{(aq)}$) in a test-tube and with caution, add 10 or so drops of freshly prepared chlorine water ($Cl_{2(aq)}$). The brown /orange colour indicates that one of the products of the reaction is iodine ($I_{2(aq)}$). This can be further tested by adding 5 mL of dichloroethane, stoppering and shaking. The dichloroethane layer turns pink/purple in the presence of iodine.

(i) Write out the total equation for the reaction.

(ii) From this total equation, deduce the two redox half-equations.

(iii) Give the net ionic equation.

(iv) Which is the oxidation 1/2-equation and which is the reduction 1/2-equation?

(v) What are the spectator ions in this reaction?

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Procedure Mini-Experiment 3

Into a clean test-tube, place about 5 mL of a **freshly** prepared solution containing iron(II) ions ($Fe^{2+}_{(aq)}$).

Into a second clean test-tube, place about 5 mL of a solution containing iron(III) ions ($Fe^{3+}_{(aq)}$).

To each solution add 0.1 mol L^{-1} sodium hydroxide ($NaOH_{(aq)}$) until a colour change is observed. Record your observations, particularly noting the colours of the precipitates. The insoluble products are iron(II)hydroxide and iron(III)hydroxide respectively.

Take the tube which initially contained the iron(II) ions in solution and shake it vigorously to allow oxygen from the air to mix with the contents. Shake until no further colour changes can be seen.

The half-equations for the observed reactions are:
$$Fe(OH)_{2(s)} \quad + \quad OH^-_{(aq)} \quad \rightarrow \quad Fe(OH)_{3(s)} \quad + \quad e^-$$

$$O_{2(g)} \quad + \quad 2H_2O_{(l)} \quad + 4e^- \quad \rightarrow \quad 4OH^-_{(aq)}$$

(i) Which is the oxidation 1/2-equation and which is the reduction 1/2-equation?

(ii) What is the balanced net ionic equation?

(iii) What is the oxidising agent?

(iv) What is the reducing agent?

Procedure Mini-Experiment 4

Into a clean test-tube, place about 5 mL of a **freshly** prepared aqueous solution of sulfur dioxide ($SO_{2(aq)}$).

To this solution add dropwise, 0.1 mol L^{-1} acidified potassium dichromate solution until no further colour change is observed.

The sulfur doixide has been oxidised to the sulfate ion $SO_4^{2-}_{(aq)}$.

The dichromate ions have been reduced to the green $Cr^{3+}_{(aq)}$ ions.

(i) Give the two redox half-equations.

(ii) Give the balanced net ionic equation.

(iii) Which is the oxidation 1/2-equation and which is the reduction 1/2-equation?

(iv) What are the spectator ions in this reaction?
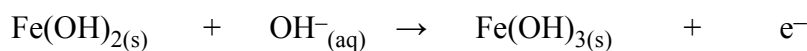
Procedure Mini-Experiment 5

(a) Into a clean test-tube, place about 5 mL of a **freshly** prepared solution containing iron(II) ions ($Fe^{2+}_{(aq)}$).

(b) Into a second clean test-tube, place about 5 mL of a solution containing iron(III) ions ($Fe^{3+}_{(aq)}$).

(c) To each solution add 5 drops of 0.1 mol L^{-1} potassium thiocyanate solution ($KSCN_{(aq)}$).

Observe the colours and recognise that $KSCN_{(aq)}$ can be used as an indicator for $Fe^{3+}_{(aq)}$.

(d) To another clean test-tube, add a further 5 mL of a **freshly** prepared solution containing iron(II) ions ($Fe^{2+}_{(aq)}$).

(e) To this solution add 10 to 15 drops of an acidified solution of potassium permanganate ($KMnO_{4(aq)}$).

(f) Now add 5 drops of 0.1 mol L^{-1} potassium thiocyanate solution ($KSCN_{(aq)}$) indicator.

        (i) What has happened to the $Fe^{2+}_{(aq)}$ ions?

        (ii) What has happened to the permanganate ions?

        (iii) Give the two redox half-equations.

        (iv) Give the balanced net ionic equation.

        (v) Which is the oxidation 1/2-equation and which is the reduction 1/2-equation?

        (vi) What are the spectator ions in this reaction?

Discussion:

With each of the 5 experiments performed,
(a) what is the similar feature of each oxidation reaction?
(b) what is the similar feature of each reduction reaction?
(c) what link must there always be between the oxidation half-equation and the reduction half-equation?

Conclusion:

Give a brief (2-3 lines only) summary of the information you gained from these 5 experiments.

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CHEMISTRY (LEVEL 4C)
EXPERIMENT 2.
PERCENTAGE PURITY OF IRON(II) SULFATE

AIM:

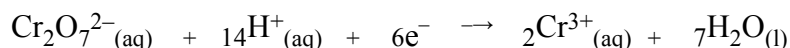To determine by volumetric analysis, the % purity (by mass) of a sample of supposedly iron(II) sulfate $FeSO_4$.

PROCEDURE:

(i) You are supplied with a solution of potassium dichromate which has been prepared and standardised as having a molarity of 0.0167 mol L^{-1}.

(ii) Prepare and fill a burette with this solution of potassium dichromate.

(iii) Into a clean dry conical beaker weigh out between 1.0 and 1.2g of the impure iron(II)sulfate crystals. Record the *accurate* mass of the crystals.

(iv) Dissolve the crystals in approximately 50 mL of 2.0 mol L^{-1} $H_2SO_{4(aq)}$ and then add approximately 5 mL of concentrated phosphoric acid ($H_3PO_{4(aq)}$) and 5 drops of the "BDS" redox indicator provided. note: (B.D.S. = barium diphenyl sulfonate)

(v) Titrate the *whole* of the prepared impure iron(II)sulfate soution against the known molarity solution of potassium dichromate from the burette. Use a magnetic stirrer to assist with the mixing during the titrating process.

(vi) The end-point is shown by the indicator changing from emerald green to deep purple with one drop.

(vii) Record the end-point volume.

(viii) If time permits, repeat the experiment with another weighed sample of impure iron(II) sulfate.

CALCULATIONS:

The two half-equations for the reaction occurring are:

$$Cr_2O_7^{2-}{}_{(aq)} \ + \ 14H^+{}_{(aq)} \ + \ 6e^- \ \longrightarrow \ 2Cr^{3+}{}_{(aq)} \ + \ 7H_2O_{(l)}$$

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

Firstly, find the balanced net redox reaction and then complete your calculations by following the steps listed below.

mass of impure $FeSO_{4(s)}$ crystals =   g

volume of potassium dichromate solution used ($K_2Cr_2O_{7(aq)}$) =   mL

molarity of potassium dichromate solution used ($K_2Cr_2O_{7(aq)}$) = 0.0167 mol L^{-1}

Thus   n($K_2Cr_2O_7$) used   =   mole

therefore,   n(Fe^{2+}) reacting   =   mole

i.e. n($FeSO_4$) present originally =   mol

$M(FeSO_4) = 152$ g mol $^{-1}$

i.e. mass of $FeSO_4$ present originally   =   g

Thus the purity of the original sample of $FeSO_4$ =   % (by mass)

DISCUSSION OF RESULTS:

Your laboratory report should include comments on:

(a) sources of error.

(b) assumptions made.

(c) possible improvements.

(d) other relevant points.

CONCLUSION:

Give a brief (2-3 line) summary of the information gained.

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CHEMISTRY (LEVEL 4C)
EXPERIMENT 3.
PERCENTAGE IRON IN STEEL WOOL

AIM: To determine by volumetric analysis, the % by mass of iron in steel wool.

PROCEDURE:

PART 1. "DISSOLVING THE STEEL WOOL"

(i) weigh out accurately approximately 0.2 g of steel wool into a clean dry 250 mL conical beaker. Record the mass to the nearest milligram. Don't allow any trace of steel wool fibres to come in contact with the electronic balance!

(ii) To the steel wool in the beaker add about 30 mL of 2.0 mol L^{-1} $H_2SO_{4(aq)}$ and then warm the mixture on a hot plate or bunsen burner until all the steel wool has dissolved. Note that some small black specks of carbon may remain.

(iii) At this stage the iron has been oxidised by the dilute sulfuric acid to $Fe^{2+}_{(aq)}$ and is in the form of pale green $FeSO_{4(aq)}$.

i.e. $$Fe(s) \quad + \quad 2H^+_{(aq)} \quad \rightarrow \quad Fe^{2+}_{(aq)} \quad + \quad H_{2(g)}$$
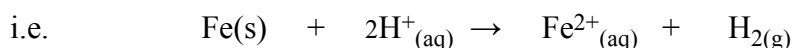
PART 2. OXIDATION OF IRON(II)

(i) You are supplied with a solution of potassium dichromate which has been prepared and standardised as having a molarity of 0.0167 mol L^{-1}.

 N.B. $M(K_2Cr_2O_7)$ = 294.2 g mol^{-1}

The solution you have been given has been prepared by dissolving 4.90 g of the $K_2Cr_2O_7$ crystals per litre of solution.

(ii) Prepare and fill a clean 50 mL burette with this solution of potassium dichromate.

(iii) Prior to commencing the titration, the indicator has to be added to the solition containing the dissolved steel wool. This is done by adding to the beaker:

   : approximately 50 mL of 2.0 mol L^{-1} $H_2SO_{4(aq)}$

   : approximately 5 mL of concentrated phosphoric acid ($H_3PO_{4(aq)}$)

   : 5 drops of the "BDS" redox indicator provided.
             note: (B.D.S. = barium diphenyl sulfonate)

(iv) Titrate the *whole* of the prepared "steel wool soution" against the known molarity solution of potassium dichromate from the burette.

(v) The end-point is shown by the indicator changing from emerald green to deep purple with one drop.

(vi) Record the end-point volume.

(vii) If time permits, repeat the experiment with another weighed sample of steel wool

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CALCULATIONS:

The balanced equation for the reaction occurring is:

$$Cr_2O_7{}^{2-}{}_{(aq)} + 14H^+{}_{(aq)} + 6Fe^{2+}{}_{(aq)} \rightarrow 2Cr^{3+}{}_{(aq)} + 6Fe^{3+}{}_{(aq)} + 7H_2O_{(l)}$$
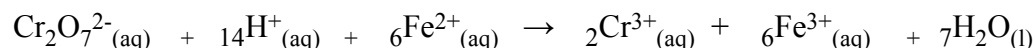
From the volume of potassium dichromate used and the solution's molarity, find:

$n(K_2Cr_2O_7)$ used   =   mole

therefore, $n(Fe^{2+})$ reacting   =   mole

i.e. $n(Fe)$ present originally =   mole

i.e. mass of Fe originally   =   g $A_r(Fe) = 55.8$

Thus, calculate the percentage of Fe in the original steel wool

DISCUSSION OF RESULTS:

Your laboratory report should include comments on:
         (a) sources of error.
         (b) assumptions made.
         (c) possible improvements.
         (d) other relevant points.

CONCLUSION:

Give a brief (2-3 line) summary of the information gained.

NOTE:
(i) Experiment 3 is very similar to experiment 2 and your teacher may decide that only one of them is to be performed.

(ii) The actual % by mass of iron in steel wool is variable but often as high as 99%. Thus even quite small errors can result in you obtaining results of greater than 100%.

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CHEMISTRY (LEVEL 4C)
EXPERIMENT 4.
OXALIC ACID CONTENT OF RHUBARB

AIM:

To determine the % by mass of oxalic acid ($H_2C_2O_4$) in rhubarb leaves by carrying out volumetric analysis. ie. using "titrations".

PROCEDURE:

The determination will involve 4 experimental stages.

> **STAGE 1**: The preparation of a primary standard solution of iron(II)ammonium sulfate $FeSO_4.(NH_4)_2SO_{4(aq)}$ of known concentration.

> **STAGE 2**: Using the solution from stage 1 to find the concentration of an unknown solution of potassium permanganate ($KMnO_{4(aq)}$).

> **STAGE 3**: Extracting the oxalic acid from the rhubarb leaves by boiling.

> **STAGE 4**: Reacting the oxalic acid from the rhubarb leaves with the standardised solution of $KMnO_{4(aq)}$.

NOTE:

As both stages 2 and 4 involve volumetric analysis ["*titrations*"] you will need to be familiar with the usage of the special glassware i.e. burettes and pipettes.

STAGE 1: PREPARATION OF THE PRIMARY STANDARD

(i) The compound iron(II) ammonium sulfate hexahydrate has the chemical formula $FeSO_4.(NH_4)_2SO_4$ **.**$6H_2O$ and has a molar mass of 392.1 g mol^{-1}.

(ii) Into a clean dry 100 mL beaker, weigh out approximately 3.9 g of the crystals and record the mass accurately.

(iii) Transfer the known mass of crystals to a clean 100 mL volumetric flask using a funnel and a wash bottle.

(iv) Before adding distilled water to the "zero" mark, add about 20 mL of 2.0 mol L^{-1} sulfuric acid solution ($H_2SO_{4(aq)}$) to the volumetric flask. This is a source of $H^+_{(aq)}$ ions.

(v) Now, carefully top-up the volumetric flask with distilled water to the zero mark. Stopper the flask and mix the solution thoroughly by tipping the flask back and forth about 10 times.

(vi) Calculate the molarity of this solution and then label the volumetric flask with your name and show the calculated concentration (use 3 sig. figs.).

(vii) This solution will be used in stage 2. This molarity is an accurate and reliable value and will not alter appreciably. It is called a "***primary standard***".

STAGE 2: STANDARDISATION OF THE UNKNOWN KMnO$_{4(aq)}$

The solution of KMnO$_{4(aq)}$ provided is approximately 0.02 mol L^{-1} but this needs to be known accurately; i.e. the solution needs to be *standardised* . This will be done by carrying out a titration involving "redox" reactions.

(i) Prepare a burette and fill it with the KMnO$_{4(aq)}$ solution. Record the 'zero' point.

(ii) Using a 20 mL bulb pipette, transfer a 20.00 mL sample of the primary standard Fe$^{2+}_{(aq)}$ solution from stage 1 into a clean 250 mL conical beaker. To this add a further 20 mL (approx.) of 2.0 mol L^{-1} sulfuric acid solution (H$_2$SO$_{4(aq)}$).

(iii) Titrate until the first permanent pink colour appears. This is the "end-point" of the titration.

(iv) Repeat the titration steps at least twice more and then determine the average volume of KMnO$_{4(aq)}$ used per titration.

(v) By establishing the balanced redox equation for the reaction, calculate the accurate concentration of the KMnO$_{4(aq)}$. This solution is now said to be "standardised".
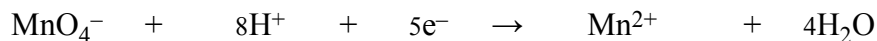

STAGE 3: EXTRACTION OF OXALIC ACID FROM RHUBARB

(i) Accurately weigh out about 20 g of fresh rhubarb leaves and cut them up into small pieces using scissors.

(ii) Place the shredded leaves in a 1L beaker and add 300 mL of distilled water. place the beaker on a hot plate and heat to boiling point.

(iii) Allow the mixture to boil for about 10 minutes during which time the leaves should be stirred and crushed with the end of a stirring rod.

(iv) During this time, the cellular structure of the leaves breaks down and releases the oxalic acid (H$_2$C$_2$O$_4$) into the water. It is quite soluble and acts as a reducing agent.

STAGE 4: OXALIC ACID DETERMINATION BY TITRATION

(i) If it is still not set up from stage 2, prepare a burette and fill it with the standardised KMnO$_{4(aq)}$ solution. Record the 'zero' point.

(ii) Using a 20 mL bulb pipette, transfer a 20.00 mL sample of the boiled rhubarb solution from stage 3 into a clean 250 mL conical beaker. To this add a further 20 mL (approx.) of 2.0 mol L^{-1} sulfuric acid solution (H$_2$SO$_{4(aq)}$).

(iii) Using a hot-plate, heat the rhubarb/sulfuric acid mixture to about 85°C. Use a clamp to hold the conical beaker.

(iv) Titrate until the first permanent pink colour appears. This is the "end-point" of the titration.

(v) Repeat the titration steps at least twice more and then determine the average volume of KMnO$_{4(aq)}$ used per titration.

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CALCULATIONS:

The redox half-equations relevant to stage 4 are:

$$H_2C_2O_4 \rightarrow 2CO_2 + 2H^+ + 2e^-$$

$$MnO_4^- + 8H^+ + 5e^- \rightarrow Mn^{2+} + 4H_2O$$

(a) Use the two half-equations to obtain a net-ionic equation.

(b) From stage 4 find the average volume of $KMnO_4$ used per titration.

(c) Knowing the $[KMnO_4]$ from stage 2, calculate the $n(MnO_4^-)$ used per titration in stage 4.

(d) Hence use the net equation to determine the $n(H_2C_2O_4)$ present per 20 mL of rhubarb extract titrated.

(e) Thus find the $n(H_2C_2O_4)$ present in the original 300 mL of rhubarb extract.

(f) Thus find the mass of $H_2C_2O_4$ present in the original 300 mL of rhubarb extract.

(g) From the mass of leaves used originally, calculate the % by mass of oxalic acid in the rhubarb leaves.

DISCUSSION OF RESULTS:

Your laboratory report should include comments on:
      (a) sources of error.

      (b) assumptions made.

      (c) possible improvements.

      (d) other relevant points.

CONCLUSION:

Give a brief (2-3 line) summary of the information gained.

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CHEMISTRY (LEVEL 4C)
EXPERIMENT 5.
INTRODUCTION TO ELECTROCHEMICAL CELLS

AIM:

To construct an electrochemical cell based on combinations of zinc and copper half-cells.

PROCEDURE:

(i) Using steel wool as an abrasive agent, prepare clean strips of both zinc and copper metal (each ~10mm x 50mm)

(ii) Into a clean 50 mL beaker place about 25 mL of 0.50 mol L^{-1} copper(II)nitrate solution $Cu(NO_3)_{2(aq)}$. Into this electrolyte immerse the copper metal strip.

(iii) Into another clean 50 mL beaker place about 25 mL of 0.50 mol L^{-1} zinc nitrate solution $Zn(NO_3)_{2(aq)}$. Into this electrolyte immerse the zinc metal strip.

(iv) Place the beakers side by side making sure that the zinc and copper trips are not touching.
(v) Using two electrical connecting wires, connect the zinc metal electrode to one terminal of a voltmeter and connect the copper electrode to the other. [The zinc should be connected to the "black" or negative terminal]. Is there any indication of current flow?

(vi) Cut a strip of filter paper about 10 cm long and 1 cm wide and immerse it into the solution of sodium nitrate ($NaNO_{3(aq)}$) supplied for making "salt-bridges".

(vii) Suspend the wet filter paper so that one end is immersed in the zinc nitrate electrolyte and the other is in the copper nitrate solution.

(viii) What is the voltage recorded on the voltmeter?

(ix) What happens to the voltmenter reading when the salt-bridge is removed?

(x) Now connect several of the electrochemical cells in the order Zn…Cu…Zn…Cu etc and record the cell voltage produced.

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DISCUSSION:

Answer the following questions in your lab. book.

(a) What EMF (voltage) was produced by this cell?

(b) What is the half-equation for the process occurring at the Zn electrode?

(c) What is the half-equation for the process occurring at the Cu electrode?

(d) What is the net equation for the cell reaction?

(e) Which electrode is the anode?

(f) Which electrode is the cathode?

(g) In which direction through the external wire do the electrons flow?

(h) Will the electrochemical cell function without a salt bridge?

(i) Explain what is occurring in the salt bridge.

(j) What is the shorthand representation for this electrochemical cell?

(k) Apart from changing the actual metals used, suggest two possible factors that will affect the voltage produced by this cell.

(l) What happened when we connected several of these electrochemical cells in series to produce a "battery"? Explain why the voltage changed.
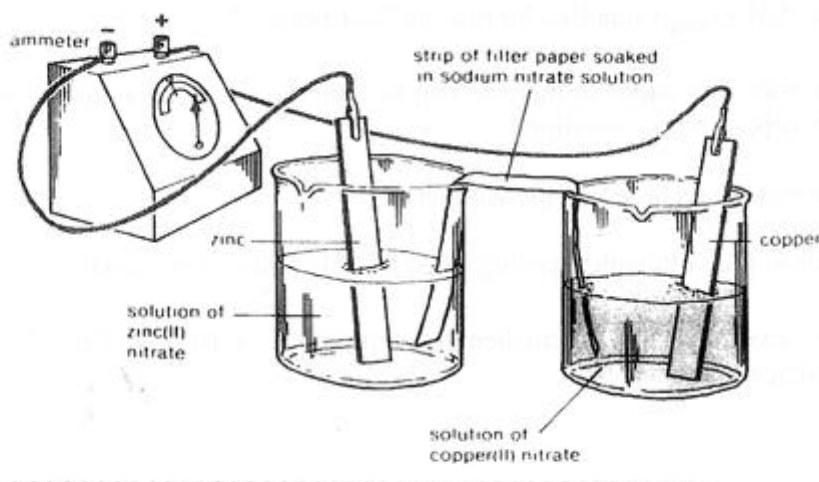

DIAGRAM OF APPARATUS:

CONCLUSION:

Give a brief summary of your findings.

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CHEMISTRY (LEVEL 4C)
EXPERIMENT 6.
COMPARING CELL VOLTAGES

AIM:

To construct a number of half-cells and then to combine them in pairs to establish the net cell EMF generated.

PROCEDURE:

For this experiment you will need to prepare six half-cells involving silver, copper, magnesium, zinc, lead and nickel.

(i) Clean all metal electrodes with steel wool before making the half-cells. Give the magnesium a particularly good cleaning to remove the oxide and nitride layer.

(ii) Into six separate 50 mL beakers pour approximately 40 mL of the 1.0 mol L^{-1} metal nitrate electrolyte. Label each beaker accordingly.

(iii) Into each aqueous electrolyte, immerse the corresponding metal electrode so that at least 20 mm of the metal is left exposed in order to allow connection to an alligator clip. This creates a 'half-cell' which can be appropriately represented by a shorthand notation; e.g. the half-cell with the copper metal electrode immersed into aqueous copper(II)nitrate is represented by the shorthand: $Cu_{(s)}/Cu^{2+}_{(aq)}$.

(iv) You should have now prepared six half cells.

(v) Prepare a U-tube salt-bridge by filling the U-tube with $NaNO_{3(aq)}$ electrolyte and inserting small cotton-wool plugs in either opening to prevent general leakage of the electrolyte.

(vi) Combine the half-cells in **PAIRS** by connecting the metal electrodes to a multimeter set on the D.C. voltage range 0-2 or 0-5 volts.

(vii) Insert the salt-bridge to complete the internal electrical circuit between the two beakers.

(viii) Take the EMF (voltage) reading quickly when the salt-bridge U-tube is in position. Don't leave the salt-bridge in position for too long otherwise contamination of the electrolyte may occur.

(ix) For each of the 15 combinations, record the cell EMF generated. Tabulate your results.

(x) When you have completed all these combinations, return the washed metal electrodes to their respective containers and the electrolytes to their containers too.

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DISCUSSION:

(a) Which half-cell combination gave the greatest EMF?

(b) By comparing the relative strengths of the metals as reducing agents, and the metal ions as oxidising agents (see the "Electrochemical Series") predict which combination should have given the greatest EMF.

(c) Which combination of half-cells gave the second greatest EMF? Does this result fit in with your predictions from the Electrochemical Series (ECS)? Explain.

(d) In accordance with the ECS, list the half-cells you prepared in order of their DECREASING electron attracting ability.

(e) In the copper ~ silver half-cell combination, which electrode was the anode? Show the appropriate half-cell equations and the net equation. What EMF should theoretically be produced?

(f) In the copper ~ nickel half-cell combination, which electrode was the cathode? Show the appropriate half-cell equations and the net equation. What EMF should theoretically be produced?

(g) Draw a complete and labelled diagram of the cell obtained by joining the $Zn_{(s)}/Zn^{2+}_{(aq)}$ and $Ag_{(s)}/Ag^{+}_{(aq)}$ half cells. What would be the shorthand representation for this cell?

CONCLUSION:

Give a brief summary of your findings

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CHEMISTRY (LEVEL 4C)
EXPERIMENT 7.
THE SILVER MIRROR EXPERIMENT

AIM: To silver plate a glass surface using a non-electrolytic process.

PROCEDURE:

(i) The surface that is to be silver plated needs to be *thoroughly clean*. The inside of a freshly cleaned test-tube is appropriate. Other surfaces may be tried later.

(ii) Place ~ 5 mL of 0.25 mol L^{-1} silver nitrate solution $AgNO_{3(aq)}$ in the test-tube.

(iii) Using a teat-pipette add 4 or 5 drops of 0.10 mol L^{-1} sodium hydroxide solution.
The brown precipitate that forms is silver oxide $Ag_2O_{(s)}$.

i.e. $2AgNO_{3(aq)} + 2NaOH_{(aq)} \rightarrow Ag_2O_{(s)} + 2NaNO_{3(aq)} + H_2O_{(l)}$

(iv) Now add dropwise to the test-tube 0.50 mol L^{-1} aqueous ammonia $NH_{3(aq)}$ until all the brown precipitate has redissolved. Don't add excess ammonia solution.
The precipitate has now formed a soluble complex ion $[Ag(NH_3)_2]^+_{(aq)}$.

This solution is called "Tollen's Reagent" and is used to test for organic reducing agents such as aldehydes and reducing sugars such as glucose, maltose and lactose.

(v) To the clear liquid in the test-tube, add 3 or 4 drops of the aldehyde supplied by your teacher. A suitable one would be aqueous methanal called "formalin".

(vi) Now place the test-tube in a water bath and continue heating until the silver mirror forms. The aldehyde has been oxidised to the corresponding carboxylic acid and the glass surface has acted as a catalytic surface for speeding up the reaction.

e.g. $CH_2O_{(aq)} + H_2O_{(l)} \rightarrow HCOOH_{(aq)} + 2H^+_{(aq)} + 2e^-$

$[Ag(NH_3)_2]^+_{(aq)} + e^- \rightarrow Ag_{(s)} + 2NH_{3(aq)}$

(vii) Wash the newly formed mirror with cold water and rinse **ALL** waste liquids into the sink with <u>large amounts of water</u>.

(viii) The silver mirror can be removed (if need be) by dissolving it in concentrated nitric acid $HNO_{3(aq)}$.

(ix) This experiment was first reported by the chemist von Liebig in 1835.

CAUTION:

The "Tollen's Reagent" should be made freshly each time and not stored, because on standing it forms a precipitate that is potentially explosive when dry!

APPLICATIONS:

Most standard mirrors are made this way as are the surfaces of "Thermos" flasks. You may wish to try silver plating other glass surfaces such as the internal surface of a flask or the concave surface of a watch glass.

This mirror production process relies very much on the catalytic properties of the surface used but glass works well.

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CHEMISTRY (LEVEL 4C)
EXPERIMENT 8.
ELECTROLYSIS OF POTASSIUM IODIDE

AIM:

To carry out a qualitative investigation involving the electrolysis of aqueous potassium iodide ($KI_{(aq)}$) using graphite electrodes. The products of electrolysis will be identified.

ELECTROLYTE USED:

The electrolyte used is a 0.50 mol L^{-1} aqueous solution of $KI_{(aq)}$.

PROCEDURE:

(i) Three quarters fill a "U" tube with the 0.50 mol L^{-1} aqueous solution of $KI_{(aq)}$ electrolyte as shown in the diagram on the right.

(ii) Immerse the two carbon (graphite) electrodes into the electrolyte and connect these by wires to the DC output of a power supply.

(iii) Increase the P.D. across the cell until it is around 12V. *Take great care with all electrical equipment so as to avoid any possibility of electric shock.*

(iv) Allow the cell to operate for about 15 minutes. Note that the (+) terminal connects to the ANODE of the electrolytic cell.

(v) Record all your observations in your laboratory note book.

(vi) After you disconnect the power supply carry out the following tests on the electrolysis products.

Carbon electrodes

TEST 1. Using a teat pipette, withdraw about 3-5 mL of the dark brown liquid from around the anode and place it in a test-tube. Add an equal volume of dichloroethane, stopper the test-tube and shake. Allow the denser dichloroethane layer to settle to the bottom. Record your observations.

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TEST 2. Using a teat pipette, withdraw about 3-5 mL of the clear liquid from around the cathode and place it in another test-tube. Add 3 drops of phenolphthalein indicator. Now test another sample of the cathode liquid with an equal volume of $FeCl_{3(aq)}$. Record all your observations.

DISCUSSION OF RESULTS:

(i) The brown colour around the anode is due to a complex I_3^- ion, indicating that iodine (I_2) was produced at the anode.
- write the equation for the anode half-reaction.

(ii) The phenolphthalein and iron(III)chloride tests reveal that the cathode region has an appreciable concentration of hydroxyl ions (OH^-) and the gas given off was hydrogen.
- write the equation for the cathode half-reaction.

CONCLUSION:

Give a brief summary of your findings.

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CHEMISTRY (LEVEL 4C)
EXPERIMENT 9.
ELECTROLYSIS OF COPPER(II) SULFATE
FARADAY'S ELECTROLYSIS LAWS

AIM:
To carry out a quantitative investigation involving the electrolysis of copper(II)sulfate using copper electrodes and to see if the data obtained is in agreement with the predictions of Faraday's Laws of electrolysis.

ELECTROLYTE USED:
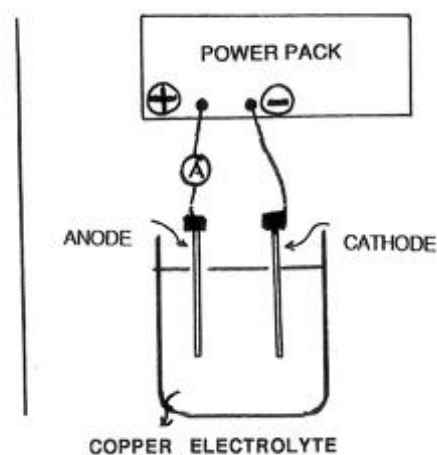The electrolyte used is a 0.50 mol/L aqueous solution of $CuSO_{4(aq)}$.

PROCEDURE
(i) Prepare two copper electrodes by cleaning them with steel wool. Keep them dry!
(ii) Label the two electrodes and then record their masses to the nearest 0.01 g using an electronic balance.
(iii) Set up the circuit as shown below using a 250 mL beaker as the electrolytic cell. (iv) Fill the beaker with about 200 mL of the copper(II)sulfate electrolyte.
(v) The copper anode in the cell is the electrode connected to the **POSITIVE** terminal of the power-pack and the cathode is connected to the **NEGATIVE** terminal. Ensure that the two copper electrodes are not touching.
(vi) Incorporate an in-series ammeter in the circuit. If available use two ammeters and they should both indicate he same current flow.
(vii) Note the starting time and switch on the power supply and quickly adjust the current flowing to 0.30 A. *Maintain the current at this value for the next hour or so.*
(viii) Allow the electrolysis reaction to continue for at least 50 minutes and then disconnect the power supply. Note the total time of cell operation.
(ix) On completion, return the copper electrolyte to the container.
(x) Rinse the two electrodes under a gentle stream of cold tap water ensuring that the perspex bar does not get wet. Dry the electrodes with acetone or use an air fan heater.
(xi) Reweigh the two electrodes and calculate the mass changes that have occurred from the initial weighings.

ELECTROLYTIC CELL SET-UP
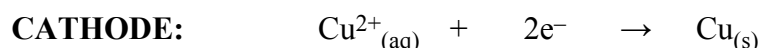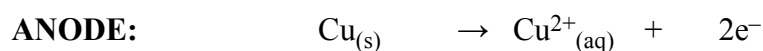
OBSERVATIONS:
(i)   Initial mass of anode   =

(ii)   Initial mass of cathode   =

(iii)   Final mass of anode   =

(iv)   Final mass of cathode   =

(v)   Current used   =

(vi)   Operating time   =

ELECTRODE HALF-EQUATIONS:

ANODE: $Cu_{(s)} \rightarrow Cu^{2+}_{(aq)} + 2e^-$

CATHODE: $Cu^{2+}_{(aq)} + 2e^- \rightarrow Cu_{(s)}$

CALCULATIONS:

(i) Calculate the mass change of the anode in grams. Why should the anode have **DECREASED** in mass?

(ii) Calculate the mass change of the cathode in grams. Why should the cathode have **INCREASED** in mass?

(iii) From the time of operation (in seconds) calculate the number of coulombs of electrical charge (Q) that passed through the cell.

$$Q = I \times t$$
I = current in amps
t = time in seconds

(iv) If the charge on one mole of electrons is 96 500 C, calculate the number of mole of electrons $n(e^-)$ that passed through the electrolytic cell.

(v) By considering the anode half-equation, calculate the n(Cu) and hence the mass of copper that should have been oxidised. Compare this result with the actual mass change recorded above.

(vi) By considering the cathode half-equation, calculate the $n(Cu^{2+})$ and hence the mass that should have been reduced. Compare this result with the actual mass change recorded above.

NOTE:

You could have determined the mass changes in the anode or cathode by directly using the Faraday Equation:

$$\text{Mass of element X electrolysed (in grams)} = \frac{(A_r(X) \times I \times t)}{(z \times 96500)} \text{ g}$$

z = ionic charge

DISCUSSION OF RESULTS:

In your discussion include the following:
(i) Reasons for the theoretical and practical mass changes differing.
(ii) Reasons for the anode and cathode mass changes not being the same.
(iii) Assumptions we have made in our calculations.
(iv) Possible improvements to the experimental procedure.
(v) Other points.

CONCLUSION:

Give a brief summary of your findings.

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CHEMISTRY (LEVEL 4C)
EXPERIMENT 10.
NICKEL PLATING EXPERIMENT

AIM To use electrolysis to nickel plate an object.
The experiment is based upon advice given by the The Hobart Plating Company for carrying out nickel plating.

ELECTROLYTE USED
The electrolyte used is an aqueous solution of the following:

300g nickel sulfate ($NiSO_4.6H_2O$) per litre

75g nickel chloride ($NiCl_2.6H_2O$) per litre

40g boric acid (H_3BO_3) per litre.

 In order to prepare this concentrated solution, the compounds need continual stirring (on a magnetic stirrer?) for 30 minutes or so at about 40°C.

 PROCEDURE
 (i) The metallic object to be electroplated needs to be thoroughly cleaned with emery paper or steel wool to ensure an adequate adhesion of the nickel metal plating.
 (ii) Set up the cell as shown below using a 100mL beaker as the electrolytic cell.
 (iii) Place a nickel metal anode in the cell and make the object to be plated, the cathode; ie. connect this object to the **negative** terminal of the power-pack.
 (iv) Incorporate an in-series ammeter in the circuit.
 (v) Switch on the power supply and adjust the current flowing to about 0.3 A.
 (vi) In order to obtain an even plating it may be necessary to rotate the object a small amount in the cell every 5 minutes or so.
 (vii) The time of operation of this electrolytic cell will be determined by the quality and thickness of the plating required.
 (viii) On completion, return the nickel electrolyte to the container and rinse the plated object under a gentle stream of water. Allow to dry.

 CONCLUSION
 Give a brief summary of the experiment

ELECTROLYTIC CELL SET-UP

POWER PACK

AMMETER

ANODE   CATHODE

NICKEL ELECTROLYTE
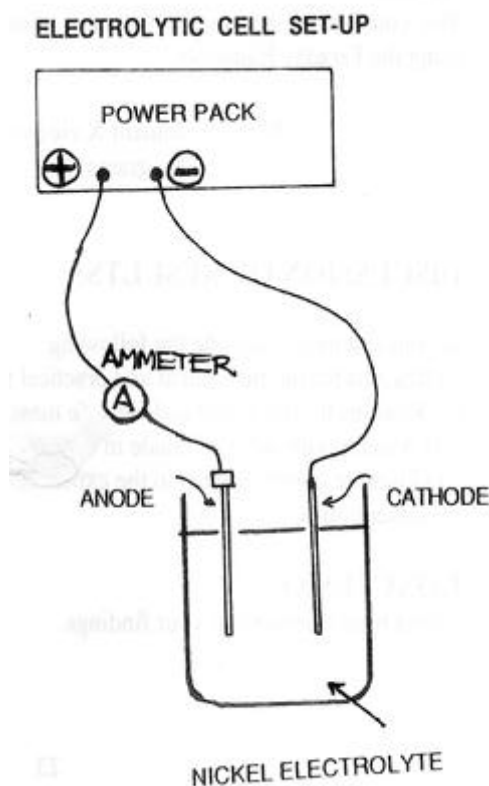
CHEMISTRY (LEVEL 4C)
EXPERIMENT 11.
INTRODUCTION TO THE CORROSION OF IRON

AIM:
To place clean iron nails in a number of different chemical environments so as to investigate the conditions necessary for corrosion.

PROCEDURE:
(a) Using steel wool, clean five iron nails and place one in each of the first five test-tubes described below.

TEST-TUBE 1:
In this test-tube place a few granules of the drying agent anhydrous calcium chloride $CaCl_{2(s)}$. Place the nail in the test-tube and stopper it tightly. The drying agent will remove water vapour from the air in the test-tube.

TEST-TUBE 2:
Boil some distilled water for a minute or so and then place enough in this test-tube to cover the nail completely. Put a thin layer of paraffin oil on top of the distilled water to prevent air from dissolving in the water. Stopper the test-tube (see diagram below).

TEST-TUBE 3:
Place enough distilled water in this test-tube to approximately cover **half** the nail. Do not stopper the test-tube.

TEST-TUBE 4:
Place enough salt-water 0.50 mol L^{-1} $NaCl_{(aq)}$ in this test-tube to approximately cover **half** the nail. Do not stopper the test-tube.

TEST-TUBE 5:
Place enough dilute hydrochloric acid $HCl_{(aq)}$ in this test-tube to approximately cover **half** the nail. Do not stopper the test-tube.

TEST-TUBE 6:
In place of the iron nail use a piece of steel wool having about the same mass as the nail. Place enough salt-water in this test-tube to approximately cover **half** the steel wool. Do not stopper the test-tube.

(b) Number your test-tubes 1 – 6 and stand them all in a test-tube rack or in a beaker.
(c) Leave the tubes for several days and then observe the chemical changes that have occurred, if any.

DISCUSSION:
(i) Which nails have corroded (rusted) most?
(ii) What conditions are necessary for rusting to occur?
(iii) When a nail corrodes, what is the:
        (a) oxidation half-equation?
        (b) reduction half-equation?
        (c) net equation?

CONCLUSION: Give a brief summary of your findings.

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CHEMISTRY (LEVEL 4C)
EXPERIMENT 12
CORROSION OF IRON &
METALS IN CONTACT IN AGAR

AIM:
To investigate the electrochemical nature of the corrosion of iron by using an agar medium to hold the products of corrosion near where they form. The investigation will specifically look at:

(i) the effects of stress on the corrosion process.

(ii) what changes occur when different metals are in contact.

PROCEDURE:
(i) Prepare about 150 mL of agar mixture as follows. In a 250 mL beaker, heat about 150 mL of distilled water to a gentle boil.

(ii) Remove the burner and stir in about 2 g of powdered agar.

(iii) Continue heating and stirring until all the agar is dispersed evenly throughout the solution.

(iv) While the solution is still hot, add and stir in thoroughly:
    (a) 10 drops of 0.10 mol L^{-1} potassium hexacyanoferrate(III).
    (b) 10 drops of 1% phenolphthalein indicator.

(v) While the mixture is cooling prepare four clean bright nails as shown in the diagram on the right.

(vi) Use one nail as a "control" and bend a second nail with pliers until it is about to fracture. Place these two nails in a petri dish and then pour the luke warm agar mixture over the nails until they are covered to a depth of about 5 mm. Don't move the dish yet!

(vii) Twist a clean piece of copper wire around a third nail and ensure that it is making good contact along all the nail.

(viii) Twist a strip of zinc metal around a fourth nail as in the diagram on the right.

(ix) Place nails 3 and 4 in a second petri dish and make sure that they don't touch one another.
(x) Pour the luke warm agar mixture over the nails until they are covered to a depth of about 5 mm. Don't move the petri dish until the agar has set.
(xi) Cover both petri dishes with lids, label them and when the agar has set, place them in a cupboard and leave them for a few days.

DISCUSSION: (after 1 or 2 days)

In your lab book, draw diagrams of the two petri dishes showing the colours that now are present. Use a code to indicate which colour is present in each region.

(i) Iron(II) ions react with potassium hexacyanoferrate(III) to form a dark blue compound called "Prussian Blue".

(ii) Phenolphthalein turns pink in regions where there is a high $[OH^-_{(aq)}]$.

(iii) Which coloured regions in the petri dishes indicate sites where oxidation has occurred i.e. anode sites? Explain your reasoning. Write the half-equation.

(iv) Which coloured regions in the petri dishes indicate sites where reduction has occurred i.e. cathode sites? Explain your reasoning. Write the half-equation.

(v) Account for the white and brown coloured regions.

(vi) How have the copper and zinc affected the corrosion of the nails? Explain.

Copper wire   Zinc strip
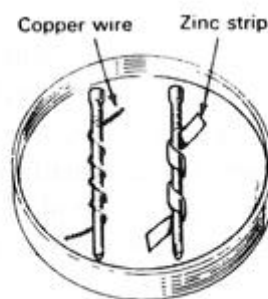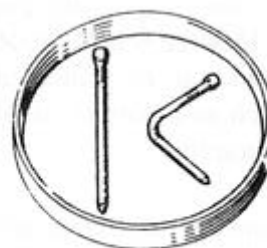
CONCLUSION:

Give a brief (2-3 line) summary of the information gained from this experiment.

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CHEMISTRY (LEVEL 4C)
EXPERIMENT 13.
APPLIED E.M.F. / IMPRESSED CURRENT

INTRODUCTION: In our theory discussions and associated experimental work we have considered a number of methods for protecting metals from corrosion. This experiment investigates another corrosion protection method that is used in particular situations where replacement of sacrificial anodes, galvanizing and painting may prove difficult or less effective. This procedure comes under the general heading of "cathodic protection" and is called"

<div align="center">

IMPRESSED CURRENT or APPLIED E.M.F.
</div>

The metallic object to be protected (usually iron/steel) is connected to the negative terminal of a D.C. supply such as a battery, whilst the positive terminal is connected to an inert electrode such as platinum or graphite.

AIM: To compare the extent of corrosion of three iron nails left in three differing corrosion environments for an extended time.

PROCEDURE:
(a) Obtain three $50 - 75$mm iron nails and ensure that they are clean and shiny by polishing them with steel wool to remove any traces of corrosion.
(b) Into three separate 50 mL (or 100 mL) beakers add salt solution (~ 0.5 mol L^{-1} $NaCl_{(aq)}$) to a depth of approximately half the length of the nails.
(c) Into beaker #1 place one of the nails and set this aside. This is the 'control' system.
(d) Into beaker #2 place a nail and a graphite electrode and using connecting wires, connect:

the **NAIL** to the positive **(+)** terminal of a **D.C.** power supply

and the **GRAPHITE** electrode to the negative **(−)** terminal.

(e) Into beaker #3 place a nail and a graphite electrode and using the same DC power supply, use two other wires to connect:

the **NAIL** to the negative **(−)** terminal of a DC power supply

and the **GRAPHITE** electrode to the positive **(+)** terminal.

(f) Turn on the power supply and using the voltmeter on the unit, operate the two cells in beakers 2 and 3 at approximately 0.5 – 1.0 volt.
Ensure that the nail and graphite are not making direct contact.
(g) After 12 hours (or more), disconnect the system and observe corrosion changes that have occurred in the three beakers if any.

DISCUSSION:
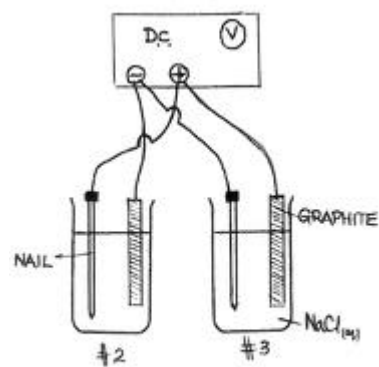Offer explanations for any variations in corrosion that may have occurred.
How do your findings fit in with theoretical expectations? Explain.
Why is the negative terminal of a car battery attached to the car's bodywork and never the positive terminal?

CONCLUSION:
Give a brief (2 – 3 lines) summary of your findings.

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CHEMISTRY (LEVEL 4C)
ANALYSIS OF VITAMIN "C" TABLETS

AIM:
To determine the vitamin C (ascorbic acid) content of tablets that supposedly contain 250 mg of vitamin C.

PROCEDURE:
(i) Find the mass of a single 250 mg vitamin C tablet. The mass may be well over 0.250 g as the tablet contains other components apart from ascorbic acid. These components are glucose, flavoring and starch to bind the tablet.
(ii) Place the tablet in a 250 mL conical beaker and add approximately 10 mL of distilled water.
(iii) Using a glass stirring rod, grind the tablet into fine particles and then add approximately a further 20 mL of distilled water.
(iv) Rinse all traces of the vitamin C off the stirring rod using no more than 20 mL of extra distilled water.
(v) Add 10 drops of concentrated $HCl_{(aq)}$ and to the vitamin C slurry and shake vigorously.
(vi) Add a small amount (0.2 g) of "vitex" indicator or use 5 drops of freshly prepared 1% starch solution instead, if vitex is unavailable.
(vii) A known standard 0.0500 mol L^{-1} solution of $I_{2(aq)}$ has been prepared for this experiment. {This solution contains by dissolving 12.69 g of solid iodine and approximately 40 g of potassium iodide in 1000 mL of aqueous solution.}
(viii) Prepare a 50 mL burette and fill it with the known 0.0500 mol L^{-1} solution of $I_{2(aq)}$.
(ix) Titrate the vitamin C solution against the 0.0500 mol L^{-1} iodine solution until a permanent blue colour is indicated by the starch. Record the volume of iodine solution used. This volume should be in the range 26 - 30 mL.
(x) Repeat the titration once more using a fresh tablet if time permits..

CALCULATIONS:
(i) Ascorbic acid (vitamin C) has the formula $C_6H_8O_6$ and it is a strong reducing agent.
(ii) $M_r(C_6H_8O_6) = 176.1$
(iii) The reaction occurring is:
$$C_6H_8O_6 \quad + \quad I_2 \quad \rightarrow \quad C_6H_6O_6 \quad + \quad 2HI$$
(iv) From your data, find the average $n(I_2)$ used per titre and hence the $n(C_6H_8O_6)$ present in each tablet.
(v) Convert this amount into grams and compare your result with the stated content of 250 mg.

DISCUSSION OF RESULTS: Your laboratory report should include:
    (a) sources of error.
    (b) assumptions made.
    (c) possible improvements.
    (d) other relevant points.

CONCLUSION: Give a brief (2-3 line) summary of the information gained.
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CHEMISTRY (LEVEL 4C)
EXPERIMENT 15.
PERCENTAGE ACETIC ACID IN VINEGAR

AIM:
The aim of this experiment is to:

(i) prepare a solution of potassium hydrogen phthalate (an acid) of known molarity in a 100 mL volumetric flask.

(ii) To titrate this potassium hydrogen phthalate solution of known molarity against a solution of sodium hyroxide $NaOH_{(aq)}$ of unknown molarity. This will enable us to determine the molarity (concentration) of the sodium hyroxide solution.

(iii) To use the $NaOH_{(aq)}$ solution to determine by titration, the concentration of acetic acid $CH_3COOH_{(aq)}$ in a vinegar solution.

PROCEDURE STEP 1:
PREPARING THE POTASSIUM HYDROGEN PHTHALATE SOLN:
(a) Into a clean dry 100 mL beaker weigh out 2.0g to 2.2g of potassium hydrogen phthalate crystals. Record the accurate mass to 3 sig. figs.

(b) Using distilled water from a "wash-bottle" and a clean funnel, wash the crystals into a clean 100 mL volumetric flask. Ensure complete transfer of all the crystals.

(c) Approximately half fill the volumetric flask with water and then dissolve the crystals by shaking the flask carefully.

(d) Now carefully fill the flask up to the 100 mL mark ensuring that the liquid level has its meniscus just on the calibration mark.

(e) Stopper the flask and then tip the flask up and down about 10 times to ensure uniform mixing. Label your flask.

CALCULATIONS (STEP 1):
(i) Given the M_r(KHPht) = 204.2 determine the number of moles of potassium hydrogen phthalate, i.e. find n(KHPht).

(ii) Remembering that these crystals are dissolved in 100 mL of solution (ie. 0.100 L) calculate the molarity of your solution i.e. find [KHPht] in mol L^{-1}

STEP 2. TITRATION OF KHPht SOLUTION AGAINST NaOH$_{(aq)}$:
(a) Wash, prepare and fill a clean burette with the unknown molarity solution of sodium hydroxide. Record in your lab. book the initial burette reading.

(b) Using a clean 20.0 mL bulb pipette, transfer 20.0 mL of the potassium hydrogen phthalate solution into a clean 250 mL conical beaker and add 4 drops of phenolphthalein indicator.

(c) Add $NaOH_{(aq)}$ from the burette until a colour change is observed from colourless to pink. Record the burette reading.

(d) Discard this final solution into the sink and wash out the flask with distilled water.

(e) Repeat steps (b) - (d) until you obtain consistent results for the end-point (ideally the readings should not vary by more than 0.2 mL)

(f) Record the average volume of $NaOH_{(aq)}$ solution used per titration.

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CALCULATIONS (STEP 2):

The potassium hydrogen phthalate can be thought of as a monoprotic acid HX and the balanced equation for the reaction occurring in this titration reaction is:

$$NaOH_{(aq)} + HX_{(aq)} \longrightarrow NaX_{(aq)} + H_2O_{(l)}$$

(i) Determine the number of mole of potassium hydrogen phthalate used in each titration. i.e. find n(KHPht) or n(HX)
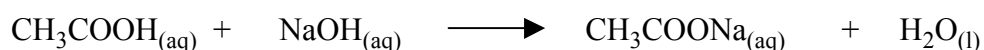
(ii) By considering the balanced equation, determine the number of mole of sodium hydroxide reacting per titration; i.e. n(NaOH)

(iii) Remembering the average volume of $NaOH_{(aq)}$ used per titration, calculate the molarity of the sodium hydroxide; i.e calculate $[NaOH_{(aq)}]$.

STEP 3. TITRATION OF SODIUM HYDROXIDE AGAINST VINEGAR:

(a) The vinegar solution needs to be diluted 10 times with distilled water before we can commence this stage. Using a clean 10 mL pipette, transfer 10.0 mL of the vinegar to a clean 100 mL volumetric flask. Carefully add distilled water up to the 100 mL mark and mix thoroughly.

(b) Continue on with the same burette filled with the now **known** molarity solution of sodium hydroxide. Record in your lab. book the initial burette reading.

(c) Using a clean 20.0 mL bulb pipette, transfer 20.0 mL of the unknown molarity vinegar solution to a clean 250 mL conical beaker and add 4 drops of phenol phthalein indicator.

(d) Add NaOH from the burette until the first permanency of pink. Record the reading.

(e) Discard this final solution into the sink and wash out the flask with distilled water.

(f) Repeat steps (c) - (d) until you obtain consistent results for the end-point (ideally the readings should not vary by more than 0.2 mL)

(g) Record the average volume of $NaOH_{(aq)}$ solution used per titration.

CALCULATIONS (STEP 3):

The balanced equation for the reaction occurring in this titration reaction is:

$$CH_3COOH_{(aq)} + NaOH_{(aq)} \longrightarrow CH_3COONa_{(aq)} + H_2O_{(l)}$$

(i) From the average volume of sodium hydroxide used and its molarity determined from part 2, find the number of mole of $NaOH_{(aq)}$ used in each titration.

(ii) By considering the balanced equation, determine the number of mole of acetic acid reacting per titration; i.e. $n(CH_3COOH)$

(iii) Remembering the volume of $CH_3COOH_{(aq)}$ used per titration is 20.0 mL calculate the molarity of the acetic acid in the diluted vinegar solution. i.e. calculate $[CH_3COOH_{(aq)}]$.

(iv) The original vinegar solution is 10 times more concentrated. Thus calculate $[CH_3COOH_{(aq)}]$ in the original vinegar.

(v) Express this concentration in (a) mol L^{-1} (b) g L^{-1} (c) % by mass

(vi) Find out what range of concentration of acetic acid is normally expected in domestic vinegars.

DISCUSSION:

Give a summary of sources of error, assumptions and possible improvements.

CONCLUSION: Give a 2-4 line summary of the information gained.

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CHEMISTRY (LEVEL 4C)
EXPERIMENT 16.
ANALYSIS OF HOUSEHOLD AMMONIA

AIM: The aim of this experiment is to:

(i) prepare a solution of sodium carbonate $Na_2CO_{3(aq)}$ (a base) of known molarity in a 250 mL volumetric flask.

(ii) To titrate this sodium carbonate solution of known molarity against a solution of sulfuric acid $H_2SO_{4(aq)}$ of unknown molarity. This will enable us to determine the molarity (concentration) of the sulfuric acid solution.

(iii) To use the $H_2SO_{4(aq)}$ solution to determine, by titration, the concentration of an unknown solution of aqueous ammonia (ammonium hydroxide) $NH_4OH_{(aq)}$.

NOTE: ammonium hydroxide ($NH_4OH_{(aq)}$) is sometimes referred to as aqueous ammonia and written as $NH_{3(aq)}$.

PROCEDURE:
STEP 1. PREPARING THE SODIUM CARBONATE SOLUTION:

(a) Into a clean dry 100 mL beaker weigh out 1.3 to 1.5 g of anhydrous sodium carbonate crystals $Na_2CO_{3(s)}$. Record the accurate mass to 3 sig. figs.

(b) Using distilled water from a "wash-bottle" and a clean funnel, wash the crystals into a clean 250 mL volumetric flask. Ensure complete transfer of all the sodium carbonate.

(c) Approximately half fill the volumetric flask with water and then dissolve the crystals by shaking the flask carefully.

(d) Now carefully fill the flask up to the 250 mL mark ensuring that the liquid level has its meniscus just on the calibration mark.

(e) Stopper the flask and then tip the flask up and down about 10 times to ensure uniform mixing. Label your flask.

CALCULATIONS (STEP 1):

(i) Determine the $M_r(Na_2CO_3)$ and then using your mass of sodium carbonate crystals determine the number of moles of sodium carbonate; i.e. find $n(Na_2CO_3)$.

(ii) Remembering that these crystals are dissolved in 250 mL of solution (ie. 0.250 L) calculate the molarity of your solution i.e. find $[Na_2CO_3]$ in mol L^{-1}

STEP 2. TITRATION OF SODIUM CARBONATE AGAINST $H_2SO_{4(aq)}$:

(a) Wash, prepare and fill a clean burette with the unknown molarity solution of sulfuric acid. Record in your lab. book the initial burette reading.

(b) Using a clean 20.0 mL bulb pipette, transfer 20.0 mL of the sodium carbonate solution into a clean 250 mL conical beaker and add 4 drops of methyl orange indicator.

(c) Add $H_2SO_{4(aq)}$ from the burette until a colour change is observed from yellow to orange/red. Record the burette reading.

(d) Discard this final solution into the sink and wash out the flask with distilled water.

(e) Repeat steps (b) $\rightarrow$ (d) until you obtain consistent results for the end-point (ideally the readings should not vary by more than 0.2 mL)

(f) Record the average volume of $H_2SO_{4(aq)}$ solution used per titration.

CALCULATIONS: (STEP 2)

The balanced equation for the reaction occurring in this titration reaction is:

$$Na_2CO_{3(aq)} + H_2SO_{4(aq)} \rightarrow Na_2SO_{4(aq)} + CO_{2(g)} + H_2O_{(l)}$$

(i) Determine the number of mole of sodium carbonate used in each titration.
i.e. find $n(Na_2CO_3)$.
(ii) By considering the balanced equation, determine the number of mole of sulfuric acid reacting per titration; i.e. $n(H_2SO_4)$
(iii) Remembering the average volume of $H_2SO_{4(aq)}$ used per titration, calculate the molarity of the sulfuric acid; i.e calculate $[H_2SO_{4(aq)}]$.

STEP 3. TITRATION OF SULFURIC ACID AGAINST $NH_4OH_{(aq)}$

(a) Continue on with the same burette filled with the now **known** molarity solution of sulfuric acid. Record in your lab. book the initial burette reading.
(b) Using a clean 20.0 mL bulb pipette, transfer 20.0 mL of the unknown molarity ammonium hydroxide $NH_4OH_{(aq)}$ solution to a clean 250 mL conical beaker and add 4 drops of methyl orange indicator.
(c) Add $H_2SO_{4(aq)}$ from the burette until the colour change occurs. Record the burette reading.
(d) Discard this final solution into the sink and wash out the flask with distilled water.
(e) Repeat steps (b) → (d) until you obtain consistent results for the end-point (ideally the readings should not vary by more than 0.2 mL)
(f) Record the average volume of $H_2SO_{4(aq)}$ solution used per titration.

CALCULATIONS: (STEP 3)

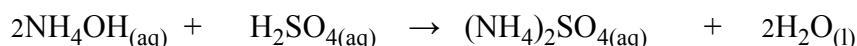The balanced equation for the reaction occurring in this titration reaction is:

$$2NH_4OH_{(aq)} + H_2SO_{4(aq)} \rightarrow (NH_4)_2SO_{4(aq)} + 2H_2O_{(l)}$$

(i) From the average volume of sulfuric acid used and its molarity determined from part 2, determine the number of mole of $H_2SO_{4(aq)}$ used in each titration. i.e. find $n(H_2SO_4)$.
(ii) By considering the balanced equation, determine the number of mole of ammonium hydroxide reacting per titration; i.e. $n(NH_4OH)$
(iii) Remembering the volume of $NH_4OH_{(aq)}$ used per titration is 20.0 mL calculate the molarity of the ammonium hydroxide solution. ie calculate $[NH_4OH_{(aq)}]$.

DISCUSSION:

Give a summary of sources of error, assumptions and possible improvements.

CONCLUSION:
Give a 2-4 line summary of the information gained from the experiment.
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CHEMISTRY (LEVEL 4C)
EXPERIMENT 17.
RELATIVE ATOMIC MASS OF MAGNESIUM

AIM:
By knowing the formula for magnesium chloride and assuming the relative atomic mass of chlorine is 35.5, we will determine the relative atomic mass of magnesium using gravimetric analysis.

PROCEDURE:
(WHERE POSSIBLE, USE **DUPLICATE** WEIGHINGS)
(i) Obtain a clean, *dry* boiling-tube (large diameter test-tube) and determine its mass accurately to the nearest 0.001 g on a milligram balance. Record this information in your laboratory note-book.
(ii) Obtain about 10 cm of magnesium ribbon and clean it carefully using steel wool ensuring that all surfaces are free of oxide and nitride layers.
(iii) Determine the mass of the magnesium metal to the nearest 0.001g. Record the mass.
(iv) **In the fume cupboard,** slowly add dropwise to the magnesium metal (using a teat-pipette), 10.0 mol L^{-1} hyrochloric acid ($HCl_{(aq)}$) until all the Mg has dissolved. Don't add more hydrochloric acid than necessary.
(v) Using a bunsen-burner with a small flame, carefully and *slowly* evaporate the remaining solution to dryness. Hold the tube with a metallic test-tube holder. The resulting white solid is magnesium chloride $MgCl_2$.
(vi) Allow the tube to cool down to room temperature (preferably in a desiccator) and then reweigh the tube and its final contents ($MgCl_2$). Record the mass to the nearest 0.001 g.

CALCULATIONS:
(a) Write a balanced chemical equation for the chemical reaction that occurred.
(b) Write down the mass of magnesium metal used in the reaction.
(c) Write down the mass of the empty boiling tube used.
(d) Write down the mass of the boiling tube plus magnesium chloride.
(e) Determine the mass of magnesium chloride product obtained in the final reaction.
(f) Determine the mass of chlorine that combined with the original mass of magnesium.
(g) Assuming the formula for magnesium chloride is $MgCl_2$ and the $A_r(Cl)$ is 35.5 calculate the $A_r(Mg)$.

DISCUSSION OF RESULTS:
(i) Find the true value for the $A_r(Mg)$ and thus calculate your % error in this experimental determination.
(ii) Suggest and discuss all possible sources of error in this experiment.
(iii) List possible improvements to the experimental procedure.
(iv) What assumptions did we make in this experimental procedure?
(v) Why do you think that this analytical procedure is called a "gravimetric" analysis?

CONCLUSION:
Give a brief (2-3 line) summary of your findings for this gravimetric determination.

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CHEMISTRY (LEVEL 4C)
EXPERIMENT 18.
ANALYSIS OF "PHOTOGRAPHIC FIXER"

AIM:

The aim of this experiment is to:

(i) prepare a solution of potassium iodate $KIO_{3(aq)}$ of known molarity.

(ii) To react this solution with excess acidified potassium iodide to release iodine.

(iii) To use the released iodine to determine, by titration, the concentration of sodium thiosulfate $Na_2S_2O_{3(aq)}$ in a sample of photographic "fixer".

PROCEDURE:

STEP 1. PREPARING THE POTASSIUM IODATE SOLUTION:

(a) Into a clean dry 100 mL beaker weigh out 1.0 to 1.1 g of potassium iodate crystals $KIO_{3(s)}$. Record the accurate mass to 3 significant figures.

(b) Using distilled water from a "wash-bottle" and a clean funnel, wash the crystals into a clean 250 mL volumetric flask. Ensure complete transfer of all the potassium iodate.

(c) Approximately half fill the volumetric flask with water and then dissolve the crystals by shaking the flask carefully.

(d) Now carefully fill the flask up to the 250 mL mark ensuring that the liquid level has its meniscus just on the calibration mark.

(e) Stopper the flask and then tip the flask up and down at least 10 times to ensure uniform mixing. Label your solution.

STEP 2. RELEASE OF IODINE FROM POTASSIUM IODATE:

(a) Using a clean 20.0 mL bulb pipette and filler, transfer 20.0 mL of the potassium iodate solution into a clean 250 mL conical beaker.

(b) To this 20.0 mL of potassium iodate solution add:

(i) approx. 20 mL of 2.0 mol L^{-1} $H_2SO_{4(aq)}$.

(ii) approx. 20 mL of 0.5 mol L^{-1} potassium **iod<u>ide</u>** KI $_{(aq)}$.

- use a graduated cylinder for measuring out these two volumes. The amounts added represent a significant excess of both $H^+_{(aq)}$ and $I^-_{(aq)}$.

(c) The reaction that occurs produces iodine $I_{2(aq)}$ according to the equation:

$$IO_3^-{}_{(aq)} \ + \ 6H^+{}_{(aq)} \ + \ 5I^-{}_{(aq)} \ \rightarrow \ 3I_{2(aq)} \ + \ 3H_2O_{(l)}$$
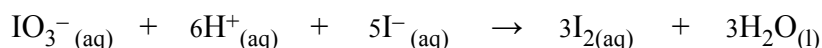
STEP 3. TITRATION OF IODINE AGAINST SODIUM THIOSULFATE:

(a) Wash, prepare and fill a clean burette with the unknown molarity solution of sodium thiosulfate (fixer) solution. Record in your lab. book the initial burette reading.
(b) Titrate the liberated iodine in the conical beaker against the sodium thiosulfate from the burette until the colour has faded to a pale yellow.
(c) Now add about 0.2g of starch indicator {"Vitex"} and continue to add the sodium thiosulfate solution from the burette until the blue colour first disappears. This indicates that all the iodine has reacted according to the equation:

$$I_{2(aq)} \quad + \quad 2\,S_2O_3{}^{2-}{}_{(aq)} \quad \rightarrow \quad 2\,I^-{}_{(aq)} \quad + \quad S_4O_6{}^{2-}{}_{(aq)}$$
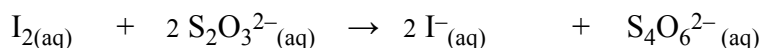
(d) record the volume of sodium thiosulfate required.
(e) Repeat Steps 2 and 3 until you obtain concordant results.
(f) Calculate the average volume of sodium thiosulfate required and use this result in your calculations.

CALCULATIONS

(i) Determine the molar mass of KIO_3.

(ii) Using your actual mass of potassium iodate crystals, determine $n(KIO_3)$ used.

(iii) Remembering that you prepared a 250.0 mL solution, calculate the concentration of your solution in moles per litre; i.e. find $[KIO_{3(aq)}]$

(iv) Find $n(IO_3{}^-{}_{(aq)})$ used in step 2 in each 20.0 mL pipetted aliquot..

(v) From the first chemical equation given, find $n(I_{2(aq)})$ liberated in step 2. Assume that all the iodate ions are reduced to iodine.

(vi) From the second chemical equation given, find $n(S_2O_3{}^{2-}{}_{(aq)})$ that reacted with the liberated iodine in step 3.

(vii) Calculate the concentration of the sodium thiosulfate solution using the data:
        (a) $n(S_2O_3{}^{2-}{}_{(aq)})$
        (b) ave. volume of $S_2O_3{}^{2-}{}_{(aq)}$ solution used.

DISCUSSION:

The "fixer" solution is expected to have a sodium thiosulfate concentration of 0.10 mol L^{-1} . What was your % difference ?
Give a summary of sources of error, assumptions and possible improvements.
By extracting the half-equations for *both* redox equations occurring, identify the oxidants and reductants involved in this experiment.

CONCLUSION:

Give a 2-4 line summary of the information gained from the experiment.

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CHEMISTRY (LEVEL 4C)
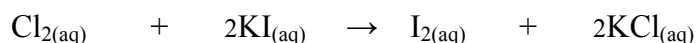# EXPERIMENT 19.
ANALYSIS OF HOUSEHOLD BLEACH

INTRODUCTION:
Domestic bleaches such as "White King", "Milton", …. are aqueous solutions of sodium hypochlorite ($NaClO_{(aq)}$). Acidifying the bleach releases chlorine into solution as $Cl_{2(aq)}$ and this can be analysed quantitatively with an "iodometric" titration using standardised $Na_2S_2O_{3(aq)}$ "fixer" solution.

AIM:
To determine the concentration of 'available' chlorine in a sample of domestic bleach.

PROCEDURE:
1. Pipette 10.00 mL of the domestic bleach into a clean 100.00 mL standard volumetric flask and dilute with distilled water up to the 100 mL mark. Mix thoroughly and label the flask as "diluted bleach".
2. Pipette a 20.0 mL aliquot of the diluted bleach into a 250 mL conical beaker and add approximately 20 mL of 2.0 mol L^{-1} $H_2SO_{4(aq)}$ and approximately 20 mL of 0.5 mol L^{-1} $KI_{(aq)}$.
3. The available chlorine reacts as follows and the brown/yellow colour indicates the presence of aqueous iodine $I_{2(aq)}$ (it's actually in the form of $I_3^{-}{}_{(aq)}$)

$$Cl_{2(aq)} \quad + \quad 2KI_{(aq)} \quad \rightarrow \quad I_{2(aq)} \quad + \quad 2KCl_{(aq)}$$

4. Titrate the released iodine against the now standardised $Na_2S_2O_{3(aq)}$ "fixer" solution from the earlier experiment. Add 'Vitex' indicator when the iodine colour fades to a pale yellow colour.
5. Repeat steps 2 – 4 until concordant results are obtained.

CALCULATIONS:
The reaction occurring in the titration step is:
$$I_{2(aq)} \quad + \quad 2S_2O_3^{2-}{}_{(aq)} \quad \rightarrow \quad 2I^{-}{}_{(aq)} \quad + \quad S_4O_6^{2-}{}_{(aq)}$$

(i) Find the number of mole of $S_2O_3^{2-}$ used on average per titre, hence the number of mole of I_2 present and thus the $n(Cl_2)$ present in the 20.0 mL of diluted bleach.
(ii) Now determine the $n(Cl_2)$ present in the original undiluted bleach.
(iii) Find the concentration of the original bleach in units of:
      (a) mol L^{-1}.
      (b) grams per litre.
(iv) Normally domestic bleaches are sold as 40 g/L available chlorine but it varies.

DISCUSSION:
Give a summary of error sources, assumptions and possible improvements.

CONCLUSION: Give a 2-4 line summary of the information gained from the experiment.

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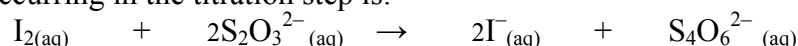
CHEMISTRY (LEVEL 4C)
EXPERIMENT 20.
PREPARATION & TESTS FOR SOME GASES

AIM: To prepare several gases and to perform identification tests for these gases.

PROCEDURE: *(wear safety glasses for all parts of this experiment)*
HYDROGEN
(i) Place a single zinc granule in the bottom of a clean test tube and using a teat pipette, add sufficient 6 mol L^{-1} $HCl_{(aq)}$ to cover the zinc.
(ii) Carefully test the gas evolved by ignition with a match.
(iii) Write and balance the chemical equations for the reaction between $Zn_{(s)}$ and $HCl_{(aq)}$ and the reaction between the evolved $H_{2(g)}$ and oxygen.

OXYGEN
(i) Place approximately 10 mL of the dilute solution of hydrogen peroxide (H_2O_2) in the bottom of a clean test tube.
(ii) Using a clean spatula, add a few crystals of potassium iodide which initially acts as a catalyst increasing the rate of decomposition of the H_2O_2.
(iii) Carefully test the gas evolved with a glowing splint.
(iv) Write and balance the chemical equations for the decomposition of the $H_2O_{2(aq)}$ into water and oxygen and the reaction between the evolved $O_{2(g)}$ and carbon in the splint..

CARBON DIOXIDE
(i) Place a single limestone chip (calcium carbonate = $CaCO_{3(s)}$) in the bottom of a clean test tube. Add sufficient 6 mol L^{-1} $HCl_{(aq)}$ to cover the calcium carbonate.
(ii) Into another clean test tube place about 5 mL of limewater which is a saturated solution of calcium hydroxide ($Ca(OH)_{2(aq)}$)
(iii) Using a clean and dry teat pipette collect a sample of the evolved carbon dioxide gas and then bubble it through the limewater solution. Observe colour changes.
(iv) Write and balance the chemical equations for the reaction between $CaCO_{3(s)}$ and $HCl_{(aq)}$ and the reaction between the evolved $CO_{2(g)}$ and limewater.

SULFUR DIOXIDE (preparation to be done in a fume cupboard!!)
(i) Place approximately 1 g of sodium sulfite powder ($Na_2SO_{3(s)}$) in the bottom of a clean test tube and using a teat pipette, add 6 mol L^{-1} $HCl_{(aq)}$ to cover the solid.
(ii) Carefully test the gas evolved by holding a piece of wet potassium dichromate paper in the neck of the test tube. Observe any colour change.
(iii) Write and balance the chemical equations for the reaction between $Na_2SO_{3(s)}$ and $HCl_{(aq)}$ and the reaction between the evolved $SO_{2(g)}$ and potassium dichromate.

HYDROGEN SULFIDE (preparation to be done in a fume cupboard!!)
(i) Place a few crystals of sodium sulfide ($Na_2S_{(s)}$) in the bottom of a clean test tube and using a teat pipette, add sufficient 6 mol L^{-1} $HCl_{(aq)}$ to cover the solid.
(ii) Carefully test the gas evolved by holding a piece of wet lead nitrate paper in the neck of the test tube. Observe any colour change.
(iii) Write and balance the chemical equations for the reaction between $Na_2S_{(s)}$ and $HCl_{(aq)}$ and the reaction between the evolved $H_2S_{(g)}$ and lead nitrate solution.

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CHEMISTRY (LEVEL 4C)
EXPERIMENT 21
BOYLE'S LAW FOR GASES

AIM:
To establish a quantitative relationship between pressure and volume for a fixed amount of gas at a constant temperature.
       i.e. to investigate Boyle's Law.

APPARATUS:
The piece of equipment used, called "Boyle's Law Apparatus", has a fixed amount of nitrogen gas inside a sealed glass tube which is calibrated in volume units of cm^3. The pressure of the gas is measured on the (red) scale in units of kPa.   (N.B. $1 \text{ kPa} = 1000 \text{ N m}^{-2}$)

PROCEDURE:
(i) Open the control tap and by using a tyre pump, increase the pressure of the gas up to a point where it is slightly greater than 350 kPa. Close the control tap.
(ii) Open the tap slowly and allow the pressure to drop until it reaches 350 kPa.
(iii) Allow 30 seconds for the oil to flow down the sides of the tube and then record the volume of gas to 3 significant figures.
(iv) Now open the control tap and allow the pressure to drop down to 330 kPa. Wait 30 seconds and again read the volume. Record your results.
(v) Repeat the above procedure, each time decreasing the pressure by 20 kPa until you are back to around 110 kPa. This should give you about 12 sets of readings as shown in the data table below.

OBSERVATIONS:

PRESSURE (kPa)	VOLUME (cm^3)	P x V ($kPa.cm^3$)

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CALCULATIONS:

(i) For each of the pressure and volume readings, calculate the value of the product $P_x V$ to three significant figures. Write this value in the third column of your data table.

(ii) Find the average value of $P_x V$ from your data table.

(iii) Find the largest value of $P_x V$ from your data table and also the smallest value.

(iv)   Calculate the % variation in your data; i.e.

$$\frac{(\text{largest} - \text{smallest})}{\text{average}} \times 100$$

(v) On graph paper plot a neat graph of pressure(P) versus volume(V) with pressure plotted on the "y" axis.

(vi) The graph should take the form of a hyperbola which suggests that the relationship is an *inverse* relationship;

i.e. $\qquad P \propto 1/V \qquad \propto = $ "proportional to"

or, $\qquad P = k(1/V) \qquad$ where $k = $ constant

or, $\qquad P \times V = k$

$\qquad\qquad 1 = $ initial conditions

i.e. $\qquad \boxed{P_1 \times V_1 = P_2 \times V_2} \qquad 2 = $ final conditions

THEORY SUMMARY:

The relationship we have established is referred to as Boyle's Law and is normally stated as:

"For a fixed amount of gas at a constant temperature, the volume of gas varies inversely with the pressure of the gas".

e.g. Doubling the pressure results in halving the volume.....etc.

DISCUSSION OF RESULTS:

Your lab report should include a brief discussion on-
  (a) assumptions made
  (b) sources of error
  (c) possible improvements

CONCLUSION:

Give a brief summary (2-3 lines) of your findings.

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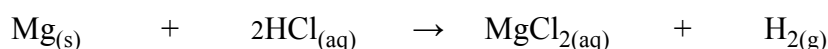
CHEMISTRY (LEVEL 4C)
EXPERIMENT 22.
MOLAR VOLUME OF H_2 GAS AT S.T.P.

AIM:

To prepare a known amount of hydrogen gas and to collect it over water so as to determine the volume occupied by one mole of $H_{2(g)}$ at S.T.P.

REACTION OCCURRING:

The hydrogen gas will be prepared by reacting a precisely known mass of magnesium metal with hydrochloric acid; i.e.

$$Mg_{(s)} \quad + \quad 2HCl_{(aq)} \quad \rightarrow \quad MgCl_{2(aq)} \quad + \quad H_{2(g)}$$

PROCEDURE:

(i) A 1.00 m length of magnesium ribbon is to be cleaned of oxide and nitride layer using steel wool. The cleaning process needs to be done in such a way so as to guarantee uniform thickness throughout the whole length.
(ii) Find the mass of the 1.00 m length of Mg. Record this mass.
(iii) Using scissors, carefully cut off a 4.00 cm length of the Mg ribbon. (The mass of this piece should be 1/25 of the mass of the metre length).
(iv) Bend the magnesium metal and encase it in a copper 'cage' as per the diagram.
(v) Pour about 20 mL of 3 mol L^{-1} $HCl_{(aq)}$ into a 50 mL gas measuring tube and then tilting the tube, carefully add distilled water to fill the tube to the top, minimising mixing of the acid and water.
(vi) Insert the copper cage with the magnesium into the gas tube and secure the cage by holding the hook in place with a 1-hole rubber stopper. (see diagram)
(vii) When properly set up the tube should contain no air bubbles and even the hole in the rubber stopper will contain distilled water.
(viii) Cover the hole in the stopper with your finger and then carefully invert the gas tube into a 1 L graduated cylinder which has been completely filled with tap water. Stand the graduated cylinder in a sink to catch the displaced water.
(ix) The acid being more dense than water will now stream down the gas tube and react with the Mg metal. The $H_{2(g)}$ will be collected in the tube.
(x) When all the Mg has reacted wait about 5 minutes for the system to reach uniform temperature. Measure and record the water temperature in the graduated cylinder.
(xi) Raise or lower the gas tube so that the liquid level inside the tube is the same as the liquid level outside. Carefully read the volume of gas and record the volume to the nearest 0.05 mL.
(xii) Repeat the experiment if time permits.
(xiii) Record the room pressure in units of mm Hg.

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DATA:

mass of 1.000 m of Mg ribbon   =

mass of 4.00 cm Mg ribbon   =

volume of gas collected   =

room temperature   =

room pressure   =

CALCULATIONS:

(i) Since the hydrogen gas was collected over water, the gas in the tube is a mixture of $H_{2(g)}$ and water vapour ($H_2O_{(g)}$). If the gas volume was measured when the liquid levels inside and outside the gas tube were the same, then the total pressure caused by these two gases is equal to the room pressure.

i.e.   Total pressure   =   pressure of $H_{2(g)}$ + pressure of $H_2O_{(g)}$

or,   pressure of $H_{2(g)}$ = P_{TOTAL} − pressure of $H_2O_{(g)}$

or;   pressure of $H_{2(g)}$ = P_{ROOM} − pressure of $H_2O_{(g)}$ .... *

(ii) The pressure exerted by the water vapour can be determined from the data table below:

TEMPERATURE (°C)	VAPOUR PRESSURE H_2O (mm Hg)
18	15.5
19	16.5
20	17.5
21	18.6
22	19.8
23	21.1
24	22.4

 (iii) Your data now includes:

Volume of (dry) H_2 gas   (V_1)   =

Pressure of H_2 gas   (P_1)   =

Temperature of H_2 gas   (T_1)   =

(iv) Using the combined gas equation, convert this volume to the volume occupied by the H_2 gas at S.T.P. Call this volume V_2 mL.

(v) From the mass of magnesium used, find n(Mg) used and hence, from the balanced equation, n(H_2) produced.

n(Mg)   =   (mass of Mg)/24.3

n(Mg)   =   n(H_2)   call this number of mole of hydrogen gas 'n'

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(vi) You now have the volume(V_2) occupied by a known number of mole of hydrogen gas (n) at S.T.P.

(vii) Thus calculate the volume occupied by ONE mole of H_2 gas at S.T.P. ....i.e.

$$\text{Practically determined molar volume of } H_2 \text{ gas at S.T.P.} = \{V_2/n\}$$

(viii) Express this answer in units of litres per mole

(ix) The theoretical figure for H_2 and in fact ALL ideal gases at S.T.P. is 22.4 L mol^{-1}.

(x) Calculate your % error.

DISCUSSION OF RESULTS:

Your lab report should include a brief discussion on-
     (a) assumptions made
     (b) sources of error
     (c) possible improvements

CONCLUSION:

Give a brief summary (2-3 lines) of your findings.

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CHEMISTRY (LEVEL 4C)
EXPERIMENT 23
EFFUSION RATES OF GASES

INTRODUCTION: Gases at the same temperature have molecules possessing the same average kinetic energy (E_K = ½mv^2). This means that gases with lower M_r values have average molecular speeds that are greater than those gases with higher M_r values. This means lower M_r gases escape faster through an opening in a container than those gases with higher M_r values.

e.g. O_2 molecules (M_r = 32.0) are 16 times heavier than H_2 molecules (M_r = 2.0) so in order to have the same kinetic energy, hydrogen molecules on average, have speeds that are 4 times greater than oxygen molecules.

AIM: To use a gas syringe to compare the rates of effusion of several gases.
(effusion = escape of gases through holes or small openings when under pressure)

PROCEDURE:
(i) Obtain a clean 100 mL gas syringe and check that the piston moves smoothly in the syringe's barrel and that there are no leaks.
(ii) Attach a 20 cm length of rubber tubing to the gas syringe and push the piston/plunger fully inwards.
(ii) Connect this rubber tube on the syringe to the glass tube extending from a plastic bag of any gas as supplied by your teacher.
(iii) By slowly withdrawing the gas syringe's piston, draw into the syringe the maximum volume of gas "X" from one of the plastic bag of gas supplied. Note the name of the gas in your lab book.
(iv) Keeping the syringe in the horizontal position, slowly push the syringe's plunger inwards until you have just 100 mL of gas in the syringe.
(v) Without delay, attach the 'effusion tube' to your syringe.
(vi) With a stop watch ready, now hold the gas syringe vertically and allow the plunger of the syringe to give the pressure to push the gas out of the hole in the effusion tube.
(vii) Record the time taken for the 100 mL of gas to pass through the hole.
(viii) Repeat this experiment with the same gas if time permits otherwise repeat the experiment with a different gas and note the time for the effusion of 100 mL to occur.

HOLE IN
EFFUSION
TUBE

TABLE OF RESULTS:

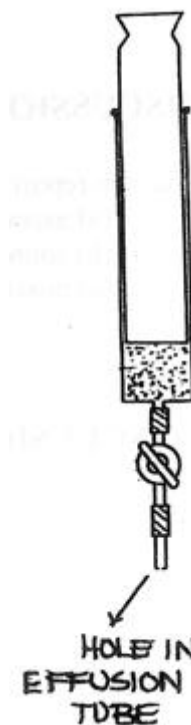| GAS	VOLUME (mL)	TIME (secs)	RATE OF EFFUSION
H_2	100 mL		mL s^{-1}
O_2	100 mL		
CO_2	100 mL		

DISCUSSION OF RESULTS:
Do the rates of effusion fit the predictions? If not why?
Is the rate of effusion of hydrogen approximately 4x the rate of effusion of oxygen as predicted?
Suggest possible sources of error.

CONCLUSION:
Give a brief summary of your findings.

CHEMISTRY (LEVEL 4C)
EXPERIMENT 24.
PERCENTAGE COMPOSITION OF L.P.G.

INTRODUCTION:
L.P.G. (liquefied petroleum gas) is used extensively as a hydrocarbon fuel for home heating, cars, barbecues, lighters and laboratories, ...
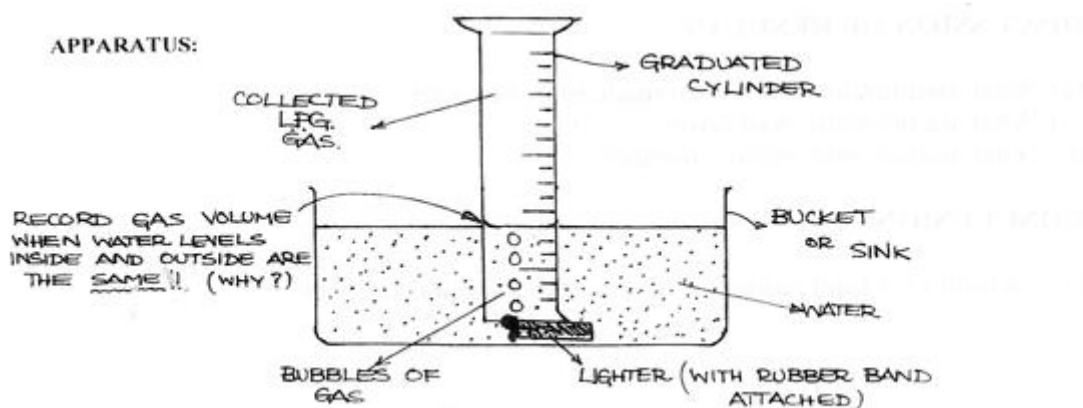LPG is a mixture of propane (C_3H_8) and butane (C_4H_{10}) with butane being the higher percentage normally. Propane and butane are both gases at room temperature and pressure but they liquefy readily under increased pressure.

AIM: To find the percentage by mass of propane in the LPG from a lighter.

PROCEDURE:
1. Find the mass of a new, dry LPG gas lighter and record the mass to the nearest $\pm$ 0.01g (centigram).
2. Completely fill a 2 L graduated cylinder with cold tap water and then carefully invert it into a sink or bucket that has been half filled with water. Ensure that no air bubbles enter the graduated cylinder during the inverting process. A piece of polythene sheet over the top of the cylinder may assist in this process.
3. Adjust the lighter's control so as to give maximum gas flow rate.
4. Place the lighter under the graduated cylinder in the water and using a stout rubber band, activate the flow of gas from the lighter. Ensure that all the gas bubbles are collected in the graduated cylinder.
5. Collect as much gas as possible but do not exceed the 2 L mark on the graduated cylinder. The lighter may run out of gas before you reach this point.
6. Stop the gas flow by removing the rubber band and record the volume of gas collected. Allow the gas to disperse into a fume cupboard. Do **not** attempt to light the gas as a major explosion may occur.
7. Record the room temperature and the barometric pressure
8. Remove the lighter from the water and dry it thoroughly before reweighing.
9. Record the final mass of the **dried** cigarette lighter.
10. Find the vapour pressure of water at the room temperature.

APPARATUS:

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CALCULATIONS:

1. Find the mass of LPG collected ('m' grams) by comparing the original mass of the lighter with the lighter's final mass.

2. Knowing the room pressure and the vapour pressure of water at room temperature, find the pressure of the LPG (p_{LPG}) in the cylinder by using the relationship that:

$$P_{TOT} = p_{LPG} + v.p._{water} \qquad P_{TOT} = room\ pressure$$

3. Now determine the number of mole of gas in the cylinder using the General Gas Equation;

$$p.V = n_{LPG}.R.T \quad i.e. \quad n_{LPG} = p.V/R.T$$

4. Let there be x grams of propane gas and thus (m – x) grams of butane gas in the mixture.
    Using $M_r(C_3H_8) = 44.0$
                      $M_r(C_4H_{10}) = 58.0$

           thus $n(C_3H_8) = x/44.0$ mole
           and $n(C_4H_{10}) = (m – x)/58.0$ mole

5. The number of mole of LPG in total will thus be the sum:

$$n_{LPG} = \{n(C_3H_8) + n(C_4H_{10})\} = \{x/44.0 + (m – x)/58.0\}$$

$$or: \quad p.V/R.T = \{x/44.0 + (m – x)/58.0\}$$

6. Solve the above equation for x and thus calculate the percentage by mass of propane in the

LPG.

DISCUSSION OF RESULTS:

(a) What assumptions have been made in this experiment?
(b) What are the sources of error?
(c) What are possible improvements?

CONCLUSION:

Give a brief (2-3 line) summary of your experimental findings.

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CHEMISTRY (LEVEL 4C) EXPERIMENT 25. INTRODUCTION TO CALORIMETRY

AIM:

To use an ethanol burner to heat a known mass of water in order to determine:

(i) the heat energy transferred to the water.
(ii) the thermal efficiency of the heat transfer process.

PROCEDURE:

(i) Using a measuring cylinder, measure out 100 mL of cold tap water and place it in a copper calorimeter.

(ii) Find the mass of an ethanol burner unit and record the mass to the nearest 0.01 g. (m_1 g)

(iii) Measure the cold water temperature with a thermometer to the nearest 0.2°C (t_1)°C

(iv) Set up the equipment as shown in the diagram below with the copper calorimeter approximately 40 mm above the burner.

(v) Light the ethanol and allow the water to heat with the flame position adjusted so as to produce the minimum amount of black smoke.

(vi) You may decide to position a screen around the system to reduce the effects of draughts on the flame.

(vii) Gently stir the water in the calorimeter with the thermometer and observe the temperature rise.

(viii) When the water temperature has reached about 80°C extinguish the flame carefully and record the maximum temperature reached to the nearest 0.2°C. This is t_2°C.

(ix) Reweigh the ethanol burner unit and record the final assembly mass to the nearest 0.01 g. (m_2 g)

can containing water

spirit lamp containing ethanol

CALCULATIONS:

(i) Using the initial and final temperatures, find the rise in temperature of the water.

i.e. $\Delta T = (t_2 - t_1)°C$

(ii) Using the initial and final assembly masses, find the mass of the candle wax burnt.

i.e. $\Delta m = (m_1 - m_2) \, g$

CALCULATIONS (continued)

(iii) Calculate the heat absorbed by the water, i.e.

$$E = \{(\text{mass of water in grams}) \times \Delta T \times 4.18\} \text{ J}$$

(iv) Using the results obtained in (ii) and (iii) above, calculate the practical heating value for the ethanol in units of joules (or kJ) per gram of ethanol.

$$\text{Practically obtained heating value} = \{E / \Delta m\} \text{ J g}^{-1} \text{ ·····································}*$$

(v) Given the chemical formula for ethanol is C_2H_6O, determine the practically obtained heating value for ethanol in units of kJ mol^{-1}.

DISCUSSION:

(a) Ethanol is known to have a theoretical heating value of 29.8 kJ g^{-1}.

(b) Use this theoretical value together with the practically obtained value to calculate the thermal efficiency of the heating process.

(c) Comment on why the process is not 100% thermally efficient by specifically discussing all possible causes of heat losses.

(d) What assumptions were made in this experiment?

(e) How might the procedure be improved so as to increase the thermal efficiency?

CONCLUSION:

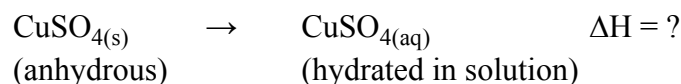Give a brief (2 - 3 line) summary of the results obtained.

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CHEMISTRY (LEVEL 4C)
EXPERIMENT 26.
DETERMINING ΔH OF SOLUTION

AIM: To determine the heat of solution for the reaction:

$$CuSO_{4(s)} \quad \rightarrow \quad CuSO_{4(aq)} \qquad \Delta H = ?$$
$$\text{(anhydrous)} \qquad \text{(hydrated in solution)}$$

PROCEDURE:
(i) Place about 10 g of the powdered hydrated crystals of copper(II) sulfate in an evaporating basin and dehydrate carefully using a bunsen burner. Gently stir the $CuSO_4 \cdot 5H_2O$ crystals with a stirring rod until the powder is nearly white.

(ii) Allow the powdered anhydrous $CuSO_4$ to cool whilst in a desiccator.

(iii) Find the mass of a *clean* copper calorimeter.

(iv) Weigh out approximately 5 g of the anhydrous copper(II) sulfate accurately into a clean, dry 100 mL beaker.

(v) Using a measuring cylinder, add 100 mL of distilled water to the copper calorimeter and record the water temperature to the nearest 0.1°C.

(vi) With continuous stirring, add the known mass of $CuSO_4$ crystals to the water and record the maximum temperature achieved.

RESULTS:

mass of copper calorimeter	=	g
mass of anhydrous $CuSO_{4(s)}$ crystals	=	g
mass of water used	=	g
initial water temperature	=	°C
final water temperature	=	°C
temperature rise(ΔT)	=	°C

CALCULATIONS:
Using: (i) specific heat of water as 4.18 J °C^{-1} g^{-1} and
(ii) specific heat of copper as 0.385 J °C^{-1} g^{-1} , calculate:

The heat absorbed by the water $\quad = E_1$ joules

The heat absorbed by the copper $\quad = E_2$ joules

Thus the heat released by forming the solution $\quad = \{E_1 + E_2\}$ J

Using the mass of $CuSO_4$ crystals, and its molar mass find:

$n(CuSO_4)$ and hence calculate the heat released in terms of kJ mol^{-1}.

DISCUSSION:
 The theoretical value is $\Delta H = -66.5$ kJ mol^{-1}

Calculate your % error and give reasons for this variation.

CONCLUSION:
 Give a brief summary of the experimental findings.

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CHEMISTRY (LEVEL 4C)
EXPERIMENT 27.
HESS'S LAW (THERMOCHEMICAL CYCLES)

AIM:

In this experiment you will measure the ΔH values for three separate reactions which make up a thermochemical cycle.

PROCEDURE:

** As there are a number of similar solutions with differing concentrations to be used in this experiment, check the solution molarities carefully before commencing each part!

PART 1: (Finding ΔH_1)

(i) Using a measuring cylinder, obtain 200 mL (ie. 200 g) of distilled water and place it in a clean polystyrene cup. This cup will act as a quite efficient calorimeter.
(ii) Using a thermometer, measure and record the water temperature to the nearest 0.1°C.
(iii) Quickly weigh out about 4.0 g of solid sodium hydroxide crystals ($NaOH_{(s)}$) or use the number of pellets that your teacher tells you approximates 4.0 g.
(iv) Pour the weighed $NaOH_{(s)}$ crystals into the distilled water and using the thermometer, stir the solution gently but continuously until all the $NaOH_{(s)}$ has dissolved.
(v) Record the highest temperature reached to the nearest 0.1°C.
(vi) Before proceeding to part 2, put the sodium hydroxide solution you have just prepared in the container labelled 0.5 mol L^{-1} $NaOH_{(aq)}$. It can be used by other classes in future lab. sessions.
(vii) Rinse out the polystyrene cup and proceed to part 2.

PART 2: (Finding ΔH_2)

(i) Repeat all the steps (i) - (v) of part 1. above except use 200 mL of 0.50 mol L^{-1} $HCl_{(aq)}$ instead of the 200 mL of distilled water.
(ii) Discard the final reaction solution into the sink, rinse the polystyrene cup and start part 3.

PART 3: (Finding ΔH_3)

(i) Measure out 100 mL of 1.0 mol L^{-1} $HCl_{(aq)}$ and place it into the polystyrene cup.
(ii) Record the temperature of the $HCl_{(aq)}$ to the nearest 0.1°C.
(iii) Using a clean measuring cylinder, now measure out 100 mL of 1.0 mol L^{-1} $NaOH_{(aq)}$.
(iv) Record the temperature of the $NaOH_{(aq)}$ in the measuring cylinder to the nearest 0.1°C.
(v) Now pour the $NaOH_{(aq)}$ solution in with the $HCl_{(aq)}$ solution in the polystyrene cup.
(vi) Stir the reactant mixture continuously with the thermometer and record the highest temperature achieved.

CALCULATIONS & RESULTS:

(i) For each part of the experiment calculate ΔT, the rise in temperature achieved.

$$\text{i.e.} \qquad \Delta T = \{T_{final} - T_{initial}\}\,°C$$

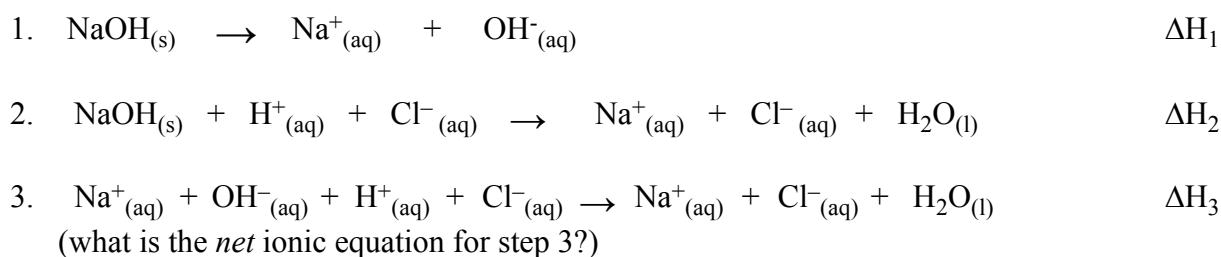Note that for part 3, use the **average** temperature of the two solutions before reaction as the initial temperature.

(ii) Assuming that in each reaction step, the reaction mixture has the same specific heat as water i.e. $4.18\ J\,g^{-1}°C^{-1}$ calculate the heat absorbed by the water; i.e.

$$E = \{(\text{grams of water heated}) \times \Delta T \times 4.18\}\ J$$

(iii) For each part, find the number of mole of NaOH used and thus determine the heats of reaction for the three parts in terms of $kJ\ mol^{-1}$ of NaOH.
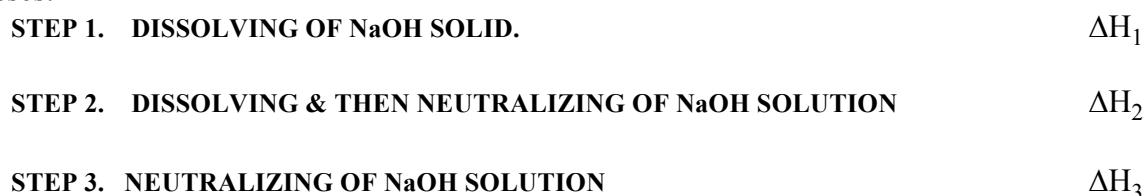
(iv) Using your data and calculations, now write the three separate thermochemical equations with actual values for ΔH_1, ΔH_2 and ΔH_3 .

The three *total* ionic equations are:

1. $NaOH_{(s)} \longrightarrow Na^+_{(aq)} + OH^-_{(aq)}$ $\qquad\qquad\qquad\qquad\qquad$ ΔH_1

2. $NaOH_{(s)} + H^+_{(aq)} + Cl^-_{(aq)} \longrightarrow Na^+_{(aq)} + Cl^-_{(aq)} + H_2O_{(l)}$ $\qquad$ ΔH_2

3. $Na^+_{(aq)} + OH^-_{(aq)} + H^+_{(aq)} + Cl^-_{(aq)} \longrightarrow Na^+_{(aq)} + Cl^-_{(aq)} + H_2O_{(l)}$ $\quad$ ΔH_3
   (what is the *net* ionic equation for step 3?)

DISCUSSION OF RESULTS:

(i) The three individual reaction steps can be thought of as involving the chemical processes:

STEP 1.   DISSOLVING OF NaOH SOLID. $\qquad\qquad\qquad\qquad\qquad$ ΔH_1

STEP 2.   DISSOLVING & THEN NEUTRALIZING OF NaOH SOLUTION $\qquad$ ΔH_2

STEP 3.   NEUTRALIZING OF NaOH SOLUTION $\qquad\qquad\qquad\qquad$ ΔH_3

These steps are related as a "thermochemical cycle", with step 2 being effectively the result of combining steps 1 and 3.

(ii) Compare the value you obtained for ΔH_2 with the combined values of $(\Delta H_1 + \Delta H_3)$

(iii) Calculate the % difference between ΔH_2 and $(\Delta H_1 + \Delta H_3)$.

(iv) Do your results reflect the expected relationship between the three ΔH values?

(v) Discuss the assumptions we have made and the sources of error that will have contributed to the % difference you obtained.

(vi) Suggest means of improving the experimental procedure.

CONCLUSION:

Give a brief (2-3) line summary of the experimental findings.

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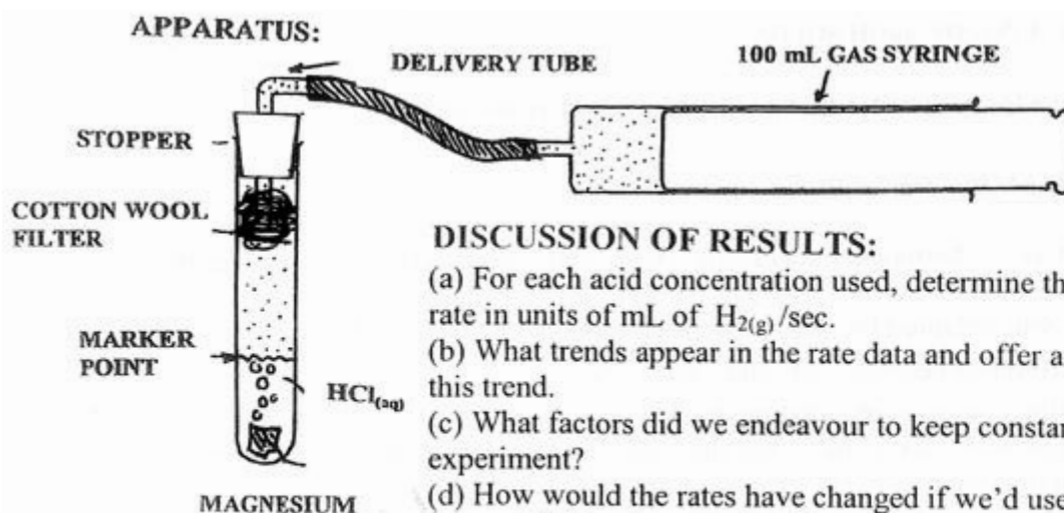
CHEMISTRY (LEVEL 4C)
EXPERIMENT 28.
EFFECT OF CONCENTRATION ON RATES

AIM:
To use a gas syringe to investigate the effect of changing concentration on the rate of the reaction between hydrochloric acid and magnesium metal.

PROCEDURE:
(i) Obtain a clean 100 mL gas syringe and check that the piston moves smoothly in the syringe's barrel and that there are no leaks.
(ii) Set up the equipment as shown in the diagram below, having the gas syringe lying horizontally on the bench and place the boiling tube in a supporting rack.
(iii) Using a measuring cylinder, add 20.0 mL of the highest molarity available $HCl_{(aq)}$ to the boiling tube. Mark the 20 mL level on the side of the boiling tube with a permanent pen.
(iv) Using scissors, cut off a 60 cm length of magnesium ribbon and clean it by removing the oxide layer using steel wool.
(v) Carefully cut the strip of magnesium ribbon into 6 separate 10 cm lengths.
(vi) Fold a 10 cm strip of Mg into a fairly tight coil. Now drop it into the hydrochloric acid and immediately insert the stopper *plus cotton wool filter* and commence the stop watch.
(vii) Record the time taken to produce 50.0 mL of hydrogen gas.
(viii) After 50.0 mL of H_2 gas has been collected remove the stopper and pour the acid contents into the container in the fume cupboard labelled "acid wastes".
(ix) Empty the gas syringe and then repeat the whole procedure using another 10 cm length of Mg ribbon and the next lowest molarity of acid available. Add the acid up to the mark and once again, record the time taken to produce 50.0 mL of hydrogen gas. (The suggested molarities of $HCl_{(aq)}$ to use are: 6.0, 4.0, 3.0, 2.0, 1.0 and 0.5 mol L^{-1})
(x) Repeat the procedure for all acid concentrations available and record your data in a table in your lab. book.

APPARATUS:

DISCUSSION OF RESULTS:
(a) For each acid concentration used, determine the average reaction rate in units of mL of $H_{2(g)}$/sec.
(b) What trends appear in the rate data and offer an explanation for this trend.
(c) What factors did we endeavour to keep constant throughout this experiment?
(d) How would the rates have changed if we'd used:
   * higher temperatures?
   * magnesium powder rather than granules?
   * a trace of aqueous Cu^{2+} ions (a catalyst for this reaction)?

CONCLUSION: Give a brief summary of your findings.

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CHEMISTRY (LEVEL 4C)
EXPERIMENT 29.
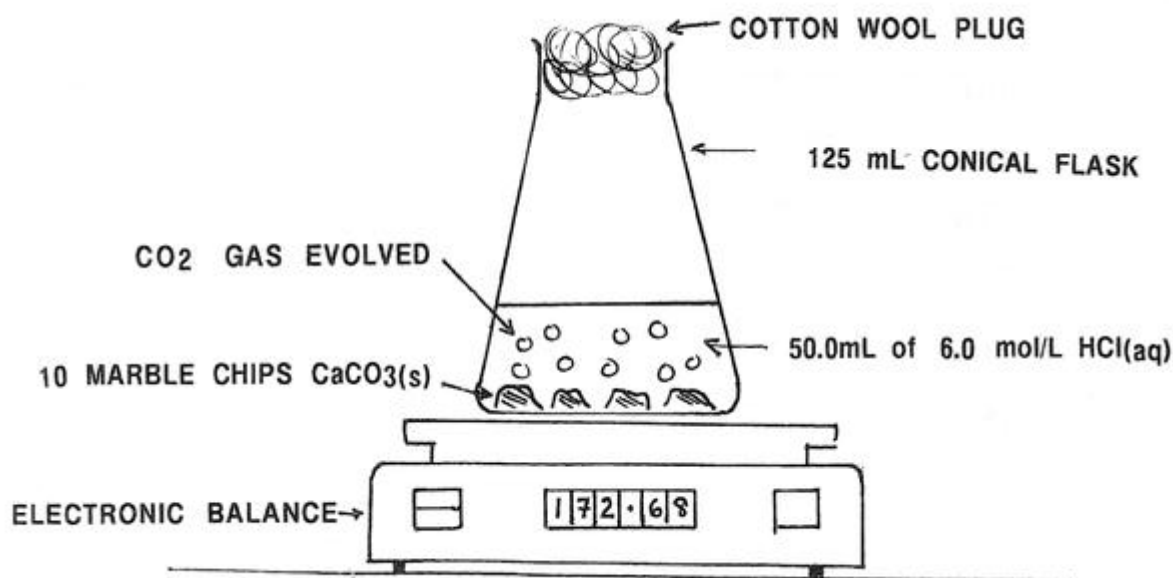# RATES OF REACTION EXPERIMENT

AIM:

To investigate the rate of the reaction between hydrochloric acid and calcium carbonate by measuring the mass of gas evolved.

PROCEDURE:

1. Measure out 50.0mL of 6.00 $molL^{-1}$ $HCl_{(aq)}$ into a 125mL conical flask. Ensure that the flask is perfectly clean, especially on the base.
2. Select sufficient marble chips ($CaCO_3$) to give a mass between 8 and 12 g. Determine their mass to ±0.01g.
3. Now place the conical flask on an electronic balance and carefully add the marble chips to the acid. Place a cotton wool plug in the flask's opening to prevent acid spray escaping!
4. Press the **TARE** button on the balance and commence taking mass readings as soon as possible. The readings become more and more negative. Why?
5. Take a reading every **half-minute** for the first 5 minutes and every minute from then on. Record these data in your laboratory book or in the table below.
6. Continue taking readings until the reaction has ceased and the mass of the system is no longer decreasing.

APPARATUS

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OBSERVATIONS:

(i) Mass of marble chips CaCO$_{3(s)}$ used = ... g

TIME (minutes)	MASS OF CO$_2$ EVOLVED (g)
0.0	0.00
05	
1.0	
1.5	
2.0	
2.5	
3.0	
3.5	
4.0	
4.5	
5.0	
6.0	
7.0	
8.0	
9.0	
10.0	
11.0	
12.0	
13.0	
14.0	
15.0	

ANALYSIS OF RESULTS:

Plot a graph of "mass of carbon dioxide evolved" against "time". (Time on the 'x' axis)
Draw a smooth curve and label your graph carefully.

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DISCUSSION OF RESULTS:

Q1. When is the reaction rate   (i) the greatest?
                                   (ii) the least?

Q2. What is the reaction rate at time
                                   (i) 1.0 minute? (use units of g of CO_2 /min)
                                   (ii) 5.0 min ?
                                   (iii) 12.0 min ?

Q3. Write the balanced chemical equation for the reaction occurring.

Q4. From your data calculate the number of moles of each reactant used. ie.
           $n(HCl_{(aq)})$ and $n(CaCO_{3(s)})$

Q5. Why does the reaction rate generally **decrease** with time? Give two reasons?

Q6. At what time did the reaction rate first reach **zero**? Explain how you deduced this.

Q7. According to your answers to Q3 and Q4. above, why did the reaction eventually stop? Explain carefully.

Q8. Your graph may show a slight reaction rate **increase** initially over the first minute or so before the general decrease in rate starts. Offer a reason for this slight initial increase in rate.

CONCLUSION:
                                 (Give a brief summary of the experimental findings)

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CHEMISTRY (LEVEL 4C)
EXPERIMENT 30.
THE "IODINE CLOCK" REACTION

INTRODUCTION

Many chemical reactions are either too fast or too slow to be studied easily in the laboratory.

However, this particular "clock" reaction is one that is easily studied and has intermediate speeds which can be quite easily altered.

The actual chemical reactions occurring are quite complex but it is convenient to regard it as a simple reaction between aqueous solutions of "A" and "B".

$$\text{i.e.} \quad A_{(aq)} \quad + \quad B_{(aq)} \quad \rightarrow \quad \text{dark blue product}$$
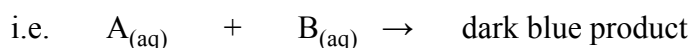
"A" is actually a solution of potassium iodate $KIO_{3(aq)}$ and solution "B" contains hydrogen sulphite ions ($HSO_3^-{}_{(aq)}$) and starch indicator. The reaction between iodate ions and hydrogen sulfite ions produces iodine ($I_{2(aq)}$) which *immediately* reacts with the $HSO_3^-{}_{(aq)}$ ions to form colourless iodide ions.

When all of the $HSO_3^-{}_{(aq)}$ ions have been used up, the iodine ($I_{2(aq)}$) forms permanently and is indicated by the starch turning dark blue.

You are required to hypothesise (propose a reasonable idea) as to the effect on the rate of the "clock reaction" of changing the:

(i) concentration of a reactant, e.g. "A"$_{(aq)}$

(ii) temperature at which the reaction is carried out.

PART 1
THE EFFECT OF CONCENTRATION ON THE RATE

(i) One member of your team should be assigned as person "A" responsible for the measuring out of solution "A". This person should have a bottle of solution "A", a 10 mL graduated cylinder, a teat-pipette and distilled water.

(ii) The other member of your team should be assigned as person "B" responsible for the measuring out of solution "B". This person should have a bottle of solution "B", a 10 mL graduated cylinder and a teat-pipette.

(iii) Don't allow the glassware for measuring out the two solutions to be interchanged!

(iv) In this experiment we will only alter the concentration of solution "A" i.e. all other factors (concentration of "B", temperature, timing procedure, mixing method,....) are to be kept constant.

(v) Firstly, person A should measure out 10.0 mL of solution "A" and person B measure out 10.0 mL of solution "B".

(vi) Pour the two solutions simultaneously into a clean 100 mL beaker and commence timing with a stop-watch from when the two solutions first mix.

(vii) Give the beaker two gentle swirls and place it on a piece of white reflective paper.

(viii) At the very first sign of blue colour stop the timing and record your result (t_1).

(ix) Discard the blue solution into the sink and wash the beaker carefully.

PART 1 (continued)

(x) Now repeat the experiment but use 9.0 mL of "A" and to this add 1.0 mL of distilled water, still giving a total volume of 10.0 mL. Use 10.0 mL of "B" for all experiments.
(xi) Use the same mixing and timing procedure as before.
(xii) Continue with the experiment but use in each step a progressively more dilute solution of "A" as shown in the table opposite.
(xiii) Record all your results in a table similar to the one shown.

RESULTS:

SOLN. "A" (mL)	WATER (mL)	SOLN. "B" (mL)	TIME (s)	RATE(mL s^{-1})
10.0	0.0	10.0	t_1	$10.0/t_1$
9.0	1.0	10.0	t_2	$10.0/t_2$
8.0	2.0	10.0	t_3	$10.0/t_3$
7.0	3.0	10.0	etc	etc
6.0	4.0	10.0		
5.0	5.0	10.0		
4.0	6.0	10.0		
3.0	7.0	10.0		
2.0	8.0	10.0		
1.0	9.0	10.0		

DISCUSSION OF RESULTS:

(a) Plot "graph 1" of the relative concentration of "A" against the 'time'. Show the relative concentration of "A" as 1.0, 0.90, 0.80, 0.70,......etc and time in seconds on the 'y' axis.
(b) Plot "graph 2" of the relative concentration of "A" against the 'rate'. The rate for each separate experiment is given as (10.0 mL of B)/time with the rate units being mL. s^{-1}. The rate goes on the 'y' axis.
(c) What information about the effect of concentration on the rate can be established from your graphical analysis? Explain.

PART 2:
THE EFFECT OF TEMPERATURE ON THE RATE:

(i) As in part 1 use different glassware for measuring out the two solutions but for each temperature use 10.0 mL of "A" and 10.0 mL of "B".
(ii) Immerse the two 10 mL graduated cylinders an ice bath or water bath to lower or raise the temperatures of solutions A and B before mixing.
(iii) Your teacher will assign two or three temperatures for you to use (between 0°C and 45°C) and all time results from the class will be used to obtain a data table.
i.e.

SOLN. "A" (mL)	SOLN. "B" (mL)	TEMP(°C)	TIME (s)	RATE(mL s^{-1})
10.0	10.0			

DISCUSSION/ CONCLUSION

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CHEMISTRY (LEVEL 4C)
EXPERIMENT 31.
CATALYSIS & INTERMEDIATE COMPOUNDS

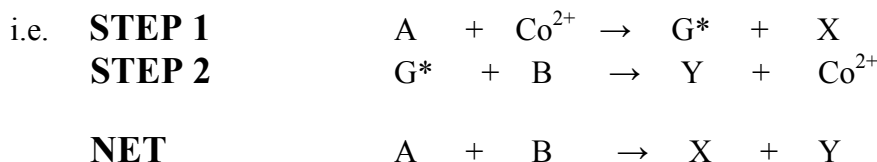**AIM:** To investigate the formation of an intermediate compound during the catalytic decomposition of potassium sodium tartrate. $KNaC_4H_4O_6.4H_2O$.
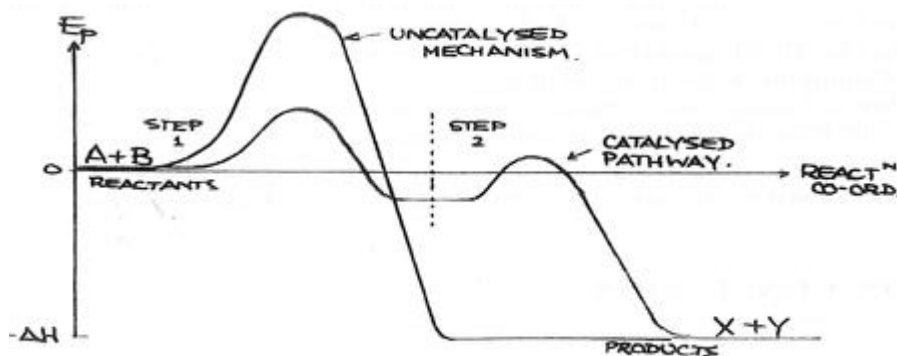
PROCEDURE:
(i) Obtain a clean 400 mL beaker and add approximately 50 mL of distilled water.
(ii) To the water add approximately 3 g of solid potassium sodium tartrate and heat the solution on a hot plate incorporating a magnetic stirrer.
(iii) Check the temperature with a thermometer and when the solution reaches $70^{o}C$ pour into the beaker 20 mL of the "20 vol" hydrogen peroxide solution. Observe carefully. If the decomposition reaction is occurring this will be indicated by the release of a gas (carbon dioxide).
 (iv) Using a spatula, now add a few grains of the catalyst, cobalt(II)chloride. Observe carefully and note all colour changes.

DISCUSSION:
* Prior to adding the catalyst, the reaction between potassium sodium tartrate and hydrogen peroxide is occurring but so slowly as to be difficult to observe.
* The addition of the catalyst causes a dramatic increase in reaction rate as evident through the rapid release of CO_2 and O_2 gases.
* The change in colour from the original pink $Co^{2+}_{(aq)}$ form to green is due to the formation of the intermediate compound cobalt tartrate.
* This green intermediate compound (cobalt tartrate = G*) is then oxidised and the green colour gradually disappears and thus the original catalyst is reformed.
*

i.e.   **STEP 1**   A   + Co^{2+} $\rightarrow$ G*   +   X
       STEP 2   G*   + B $\rightarrow$ Y   + Co^{2+}

       NET   A   + B $\rightarrow$ X   +   Y

A possible E_p diagram for the uncatalysed and catalysed reactions is shown below.

CONCLUSION:
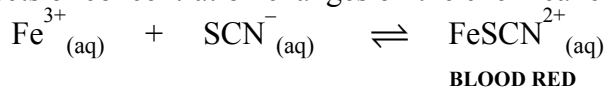Give a brief summary of your findings.
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CHEMISTRY (LEVEL 4C)
EXPERIMENT 32.
INTRODUCTION TO CHEMICAL EQUILIBRIUM

AIM:
To investigate the effects of concentration changes on the chemical equilibrium system:

$$Fe^{3+}_{(aq)} \quad + \quad SCN^-_{(aq)} \quad \rightleftharpoons \quad FeSCN^{2+}_{(aq)}$$
$$\text{\textbf{BLOOD RED}}$$

In this reaction between the yellow $Fe^{3+}_{(aq)}$ ions and the colourless $SCN^-_{(aq)}$ ions above, the product is the iron(III)thiocyanato 2+ ion which has the formula $FeSCN^{2+}_{(aq)}$ and is easily recognized by the fact that it has a **BLOOD** red colour!

PROCEDURE:
(i) Pour about 25 mL of 0.0020 mol L^{-1} $KSCN_{(aq)}$ and 25 mL of distilled water into a clean 250 mL beaker.

(ii) Using a dropping pipette, add about 5-6 drops of 0.20 mol L^{-1} $Fe(NO_3)_{3(aq)}$. Mix the resulting solution with a stirring rod and describe the products.

(iii) Now pour equal amounts of this red solution into 4 numbered petri dishes which should then be placed on a white reflective background.

(iv) **DISH #1**: this dish will be used as a standard for reference.

DISH #2: add 2-3 small crystals of solid KSCN.

DISH #3: add 2-3 small crystals of solid $Fe(NO_3)_{3(s)}$.

DISH #4: add 5-10 small crystals of solid disodium hydrogen orthophosphate $Na_2HPO_{4(s)}$

(v) For dishes 2, 3 and 4, describe the effect of the imposed change on the position of equilibrium compared with dish 1.

NB. $[X] = $ "conc. of X in mol L^{-1} "

DISCUSSION:
(i) In dish #2, which [reactant] was altered? What was the effect on the position of equilibrium?

(ii) In dish #3, which [reactant] was altered? What was the effect on the position of equilibrium?

(iii) Given that $Fe^{3+}_{(aq)}$ combines with $HPO_4^{2-}_{(aq)}$ to form a white complex ion, explain what the effect was of adding the $Na_2HPO_{4(s)}$ on:

(a) the $[Fe^{3+}_{(aq)}]$ in dish 3

(b) the position of equilibrium for the $FeSCN^{2+}_{(aq)}$ reaction.

(iv) Do the observations fit the predictions as given by Le Chatelier's Principle?

CONCLUSION:
Give a brief summary of your findings in light of the relevance of LCP to making predictions about equilibrium systems.
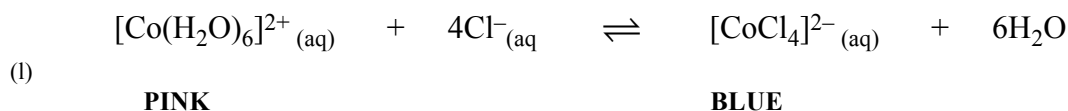
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CHEMISTRY (LEVEL 4C)
EXPERIMENT 33.
EQUILIBRIUM - COBALT COMPLEXES

AIM:
This experiment investigates the effect of changing concentrations on the equilibrium system:

(1)
$$[Co(H_2O)_6]^{2+}_{(aq)} \quad + \quad 4Cl^-_{(aq)} \quad \rightleftharpoons \quad [CoCl_4]^{2-}_{(aq)} \quad + \quad 6H_2O_{(l)}$$

PINK   **BLUE**

PROCEDURE:
(i) Set up three clean (13 x 150 mm) test-tubes in a test-tube rack.

(ii) To test-tube #1 add 5 mL of 0.2 mol L^{-1} $CoCl_{2(aq)}$ solution.

(iii) To this solution add approximately 10 mL of concentrated HCl $_{(aq)}$. Do this slowly and with caution; {wear safety glasses}. Record your observations.

(iv) Pour half the solution from test-tube #1 into a second test tube and add approximately 5 mL of distilled water. Record your observations.

(v) To the other half of the solution in test-tube #1 add 5.0 mL of 0.10 mol L^{-1} silver nitrate solution $AgNO_{3(aq)}$. Record your observations.

(vi) To the third test-tube, add 5 mL of 0.2 mol L^{-1} $CoCl_{2(aq)}$ solution. Now **slowly** pour down the side of the test-tube approximately 10 mL of acetone. Avoid mixing!

Observe the "layering effect" of the two liquids and note the colours of the regions.

NB. Acetone can form bonds with water (hydrogen bonds) and this effectively decreases the amount of water in the upper portion.

DISCUSSION:
For each part of the experimental procedure, discuss the changes occurring in light of your understanding of Le Chatelier's Principle.

APPLICATIONS:
The complex cobalt ions can be used for detecting the presence of water; eg. H_2O test paper, weather indicators and in indicators for silica gel in desiccators.

CONCLUSION:
Give a brief summary of your findings in light of the relevance of LCP to making predictions about equilibrium systems.
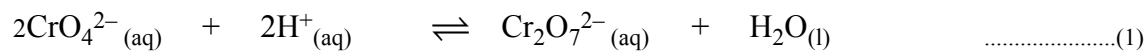
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CHEMISTRY (LEVEL 4C)
EXPERIMENT 34.
CHROMATE - DICHROMATE EQUILIBRIUM

AIM:
To investigate the effects of $[H^+_{(aq)}]$ concentration changes on the chemical equilibrium systems:

$$2CrO_4^{2-}{}_{(aq)} \quad + \quad 2H^+{}_{(aq)} \quad \rightleftharpoons \quad Cr_2O_7^{2-}{}_{(aq)} \quad + \quad H_2O_{(l)} \qquad \text{.....................(1)}$$
YELLOW   ORANGE

$$BaCrO_{4(s)} \quad \rightleftharpoons \quad Ba^{2+}{}_{(aq)} \quad + \quad CrO_4^{2-}{}_{(aq)} \qquad \text{.....................(2)}$$

PROCEDURE: (PART 1)
(i) Put approximately 5mL of 0.10 mol L^{-1} potassium <u>di</u>chromate solution, $K_2Cr_2O_{7(aq)}$ into a clean test tube and observe the orange colour associated with the dichromate ion.
(ii) Put approximately 5mL of 0.10 mol L^{-1} potassium chromate solution, $K_2CrO_{4(aq)}$ into a second clean test tube and observe the yellow colour associated with the chromate ion.
(iii) Now add one drop at a time, 1.0 mol L^{-1} $NaOH_{(aq)}$ to the orange potassium <u>di</u>chromate solution, until a colour change is observed. **Keep this solution for part 2.**
Use Le Chatelier's Principle to explain the observed effect on the equilibrium position of reaction 1 above.
(iv) Now add one drop at a time 1.0 mol L^{-1} $HCl_{(aq)}$ to the yellow potassium chromate solution, until a colour change is observed. **Keep this solution for part 2.**
Use Le Chatelier's Principle to explain the observed effect on the equilibrium position of reaction 1.

PROCEDURE: (PART 2)
(v) To the test tube from part 1 with the predominant colour of the orange dichromate ions, add a further 2-3 mL of $HCl_{(aq)}$ and then 15 drops 0.10 mol L^{-1} barium nitrate solution, $Ba(NO_3)_{2(aq)}$. Is barium dichromate soluble or insoluble? **Keep this solution for step(vii).**
(vi) To the test tube from part 1 with the predominant colour of the yellow chromate ions, add a further 2-3 mL of $NaOH_{(aq)}$ and then add 15 drops 0.10 mol L^{-1} barium nitrate solution, $Ba(NO_3)_{2(aq)}$. Is barium chromate soluble or insoluble? **Keep this solution for step (viii).**
(vii) To the solution saved from step (v) above, add the 1.0 mol L^{-1} $NaOH_{(aq)}$ solution until a change is noted.
Use Le Chatelier's Principle to explain the observed effect on the equilibrium position of reactions 1 and 2.
(viii) To the solution saved from step (vi) above, add the 1.0 mol L^{-1} $HCl_{(aq)}$ solution until a change is noted.
Use Le Chatelier's Principle to explain the observed effect on the equilibrium position of reactions 1 and 2.

DISCUSSION/CONCLUSION:
Give a brief summary of your findings in light of the relevance of LCP to making predictions about equilibrium systems.

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CHEMISTRY (LEVEL 4C)
EXPERIMENT 35.
pH TITRATION CURVE.

AIM:
To use a pH meter to detect the end-point in a standard acid-base titration between solutions of hydrochloric acid and sodium hydroxide.

PROCEDURE:
PART 1: USE OF THE pH METER.
Taking care to rinse the electrode between each reading, determine the pH of the following solutions provided:

        (a) distilled water (before boiling)
        (b) distilled water (after boiling).... why does it differ?
        (c) a dilute solution of $NaOH_{(aq)}$ of molarity about 0.10 mol L^{-1}
        (d) a dilute solution of $HCl_{(aq)}$ of molarity about 0.10 mol L^{-1}
        (e) a dilute solution of $CH_3COOH_{(aq)}$ of molarity about 0.10 mol L^{-1}

Record your results in tabular form in your lab-book.

PART 2: TITRATION USING ACID ~ BASE INDICATOR
Titrate 0.10 mol L^{-1} $NaOH_{(aq)}$ in the burette against 20.0 mL of 0.10 mol L^{-1} $HCl_{(aq)}$ in a conical beaker using 2 drops of phenolphthalein as the indicator. Repeat the titration until concordant results are obtained and record your results.

PART 3: TITRATION USING pH METER TO DETECT THE END-POINT
(i) Repeat the titration of 0.10 mol L^{-1} $NaOH_{(aq)}$ in the burette against 20.0 mL of 0.10 mol L^{-1} $HCl_{(aq)}$ in a conical beaker using a pH meter.
(ii) Take pH readings every 2.0 mL up to 1 mL before the average end-point determined in part 3 i.e. if the end-point occurred at 19.2 mL, take readings at 0, 2, 4, 6, 8, ... 18 mL of $NaOH_{(aq)}$ added.
(iii) Now take pH readings at every 0.2 mL of $NaOH_{(aq)}$ added until you are 1 mL beyond the average end-point determined in part 3.
(iv) Finally, take pH readings at every 2 mL of $NaOH_{(aq)}$ added until you are 16 mL beyond the average end-point determined in part 3.
(v) Record your results in tabular form.

DISCUSSION OF RESULTS:
(a) Plot a pH curve showing the change in pH (on the vertical axis) against volume of $NaOH_{(aq)}$ added, on the horizontal axis. Describe the shape of this acid~base titration curve.
(b) In quantitative terms, describe the change in [H$^+$$_{(aq)}$] at the end-point of a standard acid-base titration.

CONCLUSION:
Give a brief summary of the information gained in performing this experiment.
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CHEMISTRY (LEVEL 4C)
EXPERIMENT 36.
ACID-BASE STRENGTH EXPERIMENT

AIM:

To find the percentage dissociation (ionization) in samples of:

        (i) acetic acid $CH_3COOH_{(aq)}$ a weak acid.

        (ii) aqueous ammonia $NH_4OH_{(aq)}$ a weak base.

PROCEDURE:

(i) In a clean 50mL beaker, place approximately 20 mL of acetic acid. In your laboratory book, record the molarity of the acetic acid.

(ii) Using a previously calibrated pH meter, carefully measure the pH of the acetic acid and record the value.

(iii) Repeat the two steps above for the solution of aqueous ammonia provided.

CALCULATIONS:

(a) ACETIC ACID:

From the pH value obtained find the concentration of aqueous hydrogen ions in the solution using the relationship that:

$$pH = -\log[H^+_{(aq)}]$$
$$\text{i.e. } [H^+_{(aq)}] = 10^{-pH}$$

Using the molarity of the acetic acid solution, thus determine the % dissociation of the acetic acid.

(b) AMMONIA:

From the pH value obtained find the concentration of aqueous hydrogen ions in the solution using the relationship that:

$$pH = -\log[H^+_{(aq)}]$$
$$\text{i.e. } [H^+_{(aq)}] = 10^{-pH}$$

From this figure for the hydrogen ion concentration, calculate the hydroxyl ion concentration using the relationship that:

$$[H^+_{(aq)}]\,[OH^-_{(aq)}] = 1.00 \times 10^{-14} \ \ (25°C)$$

Using the molarity of the ammonia solution, thus determine the % dissociation of the ammonia solution.

DISCUSSION:

Give a summary of sources of error, assumptions and possible improvements.

CONCLUSION:

Give a 2-4 line summary of the information gained from the experiment.

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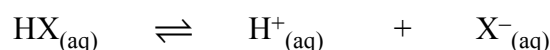
CHEMISTRY (LEVEL 4C)
EXPERIMENT 37.
DISSOCIATION CONSTANT FOR A WEAK ACID

AIM:
To obtain experimentally the dissociation constant (K_a) for the weak acid potassium hydrogen phthalate.

INTRODUCTION:
The weak acid potassium hydrogen phthalate has the formula $KHC_8H_4O_4$ with a molar mass of 204.1 g mol^{-1}. It behaves as a monoprotic acid "HX" dissociating very sparingly i.e.

$$HX_{(aq)} \rightleftharpoons H^+_{(aq)} + X^-_{(aq)}$$

PROCEDURE:
(i) Into a clean dry 100 mL beaker weigh out approximately one gram of the solid potassium hydrogen phthalate. The mass needs to be in the range 1.00 – 1.10g and known accurately to 3 significant figures. Record the mass of solid used.
(ii) Using a wash bottle and a funnel, rinse the crystals into a 100 mL volumetric flask using distilled water. Ensure complete transfer of all the crystals.
(iii) Dissolve the crystals by shaking and then make the volume up to the accurate 100 mL mark.
(iv) Transfer this known molarity solution into a clean 250 mL conical beaker and measure the solution's pH using a previously calibrated pH meter. Record the pH value.
Use at least three separate pH meters and average the readings that are within close agreement.

CALCULATIONS:
(a) From your mass of KH phthalate crystals, calculate the number of moles used and hence find the initial molarity of your solution to 3 significant figures. It should be ~ 0.05 mol L^{-1}.
(b) From your pH value, calculate the hydrogen ion concentration present in the solution after dissociation had reached equilibrium.

$$\text{N.B.} \quad [H^+_{(aq)}] = 10^{-pH}$$

(c) Now using your results from (a) and (b) above, complete an "I.C.E." diagram.

(d) Using the *equilibrium* data from your I.C.E. diagram, calculate the value of K_a for the weak acid "HX" (potassium hydrogen phthalate) at 25°C. What is the theoretical value?

DISCUSSION
The theoretical value for K_a (potassium hydrogen phthalate) at 25°C is 1.91 x 10^{-7}.
What was your % error and what were the most likely causes of this error?

CONCLUSION:
Give a 2-4 line summary of the information gained from the experiment.

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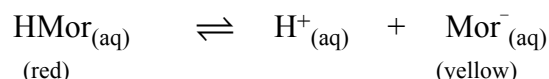
CHEMISTRY (LEVEL 4C)

EXPERIMENT 38.
K$_A$ FOR METHYL ORANGE

AIM:
To determine experimentally the approximate value of K$_a$ for the acid/base indicator methyl orange.

INTRODUCTION:
The acid/base indicator methyl orange is a weak acid having a complex organic structure.
It behaves as a monoprotic acid ("HMor") dissociating very sparingly;
 i.e.

$$HMor_{(aq)} \rightleftharpoons H^+_{(aq)} + Mor^-_{(aq)}$$
(red)   (yellow)

PROCEDURE:
(i) In a test-tube rack, set up six identical, **clean** test tubes.
(ii) To test-tube #1 add 10 teat-pipette volumes (i.e. 10 "squirts") of 0.10 mol L^{-1} HCl$_{(aq)}$.
(iii) Using a marker pen, now mark the height of this solution on each of the other 5 test-tubes.
(iv) From test-tube #1 extract **one** squirt of the solution and add it to test-tube #2. To this, now add distilled water up to the mark. Stopper the test-tube and shake it to ensure even mixing.
(v) Now, from test-tube #2 extract **one** squirt of the solution and add it to test-tube #3. To this, add distilled water up to the mark. Stopper the test-tube and shake it to ensure even mixing.
(vi) Continue this dilution process until you have the 6 test-tubes each with a progressively diluted solution of HCl$_{(aq)}$.
(vii) To each test-tube add 2 drops of methyl orange indicator and mix thoroughly.
(viii) Using a white background, observe the colour of each of the six test-tubes and attempt to identify in which tube the indicator appears to be in the intermediate colour form i.e. between the red and yellow and in the **orange** colour form.

CALCULATIONS:
(i) Each successive test-tube has an acidic solution which has been effectively diluted 10 times.
(ii) What is the concentration of hydrogen ions in each tube? (assume HCl$_{(aq)}$ is fully ionized)
(iii) What is the pH of the solution in each tube?
(iv) What is the colour of the indicator in each of the 6 test-tubes?
* Summarise your answers to these questions above in the form of a table of results.
(v) Write the equilibrium constant expression K$_A$ for methyl orange.
(vi) The intermediate colour for the indicator occurs when the concentration of the red form of the indicator is approximately the same as the concentration of the yellow form.
 i.e. intermediate colour occurs when $[HMor_{(aq)}] \simeq [Mor^-_{(aq)}]$
(vii) From this observation you can now establish an approximate value for the K$_A$.

DISCUSSION ~ CONCLUSION:
The theoretical value for K$_A$ (methyl orange) at 25°C is 2.0 x 10^{-4}.
For which types of acid/base titrations is methyl orange:
        (i) particularly suitable?
        (ii) not suitable? Why?

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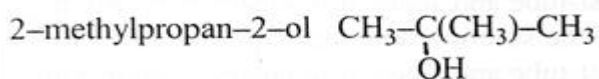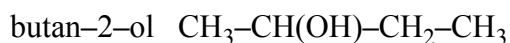
CHEMISTRY (LEVEL 4C)
EXPERIMENT 39.
REACTIONS OF ALCOHOLS

AIM:

To investigate some oxidation reactions of some primary, secondary and tertiary alcohols.

PROCEDURE: PART 1. OXIDATION OF ALCOHOLS

There are 5 different alcohols to be tested in this part. They are:

ethanol $CH_3–CH_2–OH$

butan–1–ol $CH_3–CH_2–CH_2–CH_2–OH$

butan–2–ol $CH_3–CH(OH)–CH_2–CH_3$

2–methylpropan–2–ol $CH_3–\overset{\displaystyle}{\underset{\displaystyle OH}{C}}(CH_3)–CH_3$

2–methylpropan–1–ol $CH_3–CH(CH_3)–CH_2–OH$

Draw structures and classify each of the above as either a primary, secondary or tertiary alcohol.
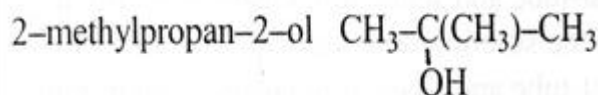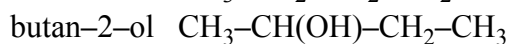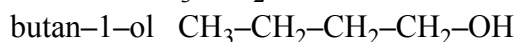(ii) Place approximately 2 mL of each alcohol in 5 separate, clean test tubes in a test tube rack.
(iii) To each alcohol add about 5 drops of the acidified solution of the oxidant potassium permanganate $(KMnO_{4(aq)})$ provided.
(iv) Stopper the tube and shake carefully for 30 seconds. Record your observations of colour changes, if any.

PROCEDURE PART 2. REACTION WITH SODIUM METAL

(i) There are 4 different alcohols to be tested in this part. They are:
    ethanol $CH_3–CH_2–OH$
    butan–1–ol $CH_3–CH_2–CH_2–CH_2–OH$
    butan–2–ol $CH_3–CH(OH)–CH_2–CH_3$

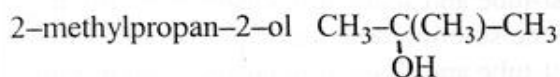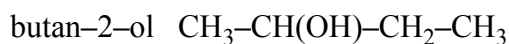2–methylpropan–2–ol $CH_3–\overset{\displaystyle}{\underset{\displaystyle OH}{C}}(CH_3)–CH_3$

(ii) Place approximately 2 mL of each alcohol in 4 separate, DRY test tubes in a test tube rack.
(iii) To each alcohol add a small piece of sodium metal (CAUTION) and observe.
(iv) Compare the different rates of reaction. The gas evolved is hydrogen.

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PART 3. REACTION WITH CONCENTRATED HYDROCHLORIC ACID

(i) There are 3 different alcohols to be tested in this part. They are:

butan–1–ol $CH_3–CH_2–CH_2–CH_2–OH$

butan–2–ol $CH_3–CH(OH)–CH_2–CH_3$

2–methylpropan–2–ol $CH_3–\underset{\underset{OH}{|}}{C}(CH_3)–CH_3$

(ii) Place approximately 2 mL of each alcohol in 3 separate, clean test tubes in a test tube rack.

(iii) To each alcohol add about 5 mL of conc. $HCl_{(aq)}$ (CAUTION) and observe.

(iv) The formation of a cloudiness (slight precipitate) indicates the formation of the corresponding alkyl chloride.

(v) Compare the different rates of reaction.

PART 4. OXIDATION OF METHANOL WITH HOT COPPER OXIDE

(i) Place about 10 mL of methanol ($CH_3–OH$) in a clean 100 mL beaker and heat the methanol until it boils. NOTE: If the methanol ignites cover the beaker with a wet sponge (or similar) to extinguish the flame.

(ii) In the meantime, heat a 1 cent or 2 cent coin until red hot by holding it with crucible tongs in the bunsen flame.

(iii) Quickly transfer the hot coin (which should now appear black) into the vapours of methanol and observe the change in surface appearance of the coin.

(iv) Cautiously note the change in odour due to the formation of methanal (HCHO)

DISCUSSION OF RESULTS:

(i) Which categories of alcohols appear to undergo oxidation?

(ii) Give the balanced equations for the oxidation reactions occurring when:
- ethanol is oxidised by excess acidified $KMnO_4$.
- butan-1-ol is oxidised by excess acidified $KMnO_4$.
- butan-2-ol is oxidised by excess acidified $KMnO_4$.

(give IUPAC names for all products formed)

(iii) Give the balanced equation for the reaction occurring when sodium metal reacts with:
- butan-1-ol   (give IUPAC names for the products formed)

(iv) Give the balanced equation for the reaction occurring when concentrated $HCl_{(aq)}$ reacts with the **tertiary** alcohol you used. (use structural formulae and name the product)

(v) Give the balanced equation for the reaction occurring when methanol is oxidised by hot $CuO_{(s)}$ to form methanal. (show the structural formulae and names for the organic compounds)

CONCLUSION:

 Give a brief summary of your findings.

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CHEMISTRY (LEVEL 4C)
EXPERIMENT 40.
"BREATHALYSER" SIMULATION

AIM:
To investigate the colour changes that occur during the oxidation of ethanol using acidified potassium dichromate.

THEORY:
Alcoholic drinks contain ethanol (CH_3–CH_2–OH) which is a reducing agent and will reduce yellow dichromate $Cr_2O_7{}^{2-}{}_{(aq)}$ ions to the green/blue coloured $Cr^{3+}{}_{(aq)}$ ions in an acidic aqueous solution. The level of colour change from yellow to green/blue can be measured with a light meter and the reading used to gauge the amount of ethanol in a driver's breath. Most states in Australia set the maximum legal limit at 0.05% i.e. 0.5 mg of ethanol per mL of blood.

PROCEDURE:
(i) Using a hot-plate, prepare a hot water bath in a 400 mL beaker, (use ~ 100 mL).
(ii) Into each of 5 separate, clean test tubes carefully add 4 mL of 0.017 mol L^{-1} $K_2Cr_2O_{7(aq)}$ solution plus 1 mL of 1.0 mol L^{-1} $H_2SO_{4(aq)}$. *Number the test tubes*.
(iii) Using a teat pipette add:

> 1 drop of ethanol to test tube #2
> 3 drops of ethanol to test tube #3
> 8 drops of ethanol to test tube #4
> 12 drops of ethanol to test tube #5

(iv) Test tube #1 will be used as a reference colour or 'control'.
(v) <u>At the same time</u>, place all 5 test tubes in a water bath at 100°C and leave to heat for two to three minutes.
(vi) Remove all 5 test tubes from the water bath and stand them in numerical order in a test-tube rack.
(vii) Using a white paper background, compare and contrast the variation in colours. Record your observations in a tabular form.

DISCUSSION OF RESULTS:
(i) Give the balanced equations for the oxidation reaction occurring when ethanol is oxidised to ethanal by acidified $Cr_2O_7{}^{2-}{}_{(aq)}$.
(ii) Explain the reasons for the colour changes that occurred.
(iii) Was there a significant variation in the final colours after heating? Suggest reasons for these colour differences.
(iv) At 20°C the reaction rate is quite slow. Why did heating increase the reaction rate?
(v) It is possible that some of the ethanal was oxidised further. What would be the resulting product? Give the appropriate chemical equation for this further reaction
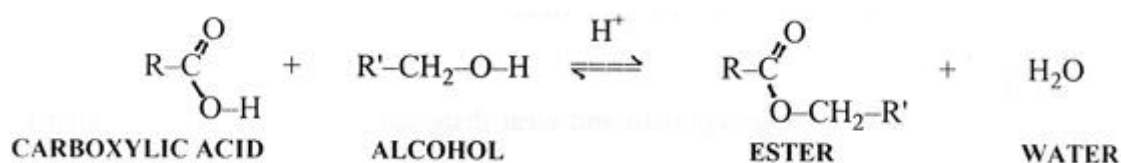
CONCLUSION:
Give a brief summary of your findings.

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CHEMISTRY (LEVEL 4C)
EXPERIMENT 41.
PREPARATION OF SOME ESTERS

AIM: To prepare a number of different esters and to identify the odours of the esters formed.

THEORY: Esterification involves the equilibrium reaction between a carboxylic acid and an

alcohol in the presence of catalyst   i.e.

PROCEDURE:
(i) Place about 100 mL of hot tap water in a 400 mL beaker and put the beaker on a hot-plate and heat the water up to near boiling point (90 - 95°C)
(ii) Each ester is prepared by adding approximately 2 mL of the alcohol and approximately 2 mL of the carboxylic acid to a clean dry test tube.
(iii) Then cautiously add 3 drops of concentrated sulfuric acid which is the catalyst for the esterification reaction.
(iv) Label the test tube with an identifying number and then place it in the hot water bath.
(v) Repeat the above steps so that you have four or five different esters being formed. Place all your labelled test tubes in the water bath and wait for about 10 minutes. (The esterification reaction is fairly slow even when catalysed at 95°C because the reaction involves breaking of covalent bonds.)
(vi) For each ester in turn, waft the emitted vapours cautiously towards your nose and check that the odour is as expected according to the table below.

ESTERS YOU MAY WISH TO PREPARE:

ALCOHOL	CARBOXYLIC ACID	ESTER	ODOUR
butan-1-ol	butanoic acid	butyl butanoate	pineapples
ethanol	butanoic acid	ethyl butanoate	strawberries
butan-1-ol	methanoic acid	butyl methanoate	raspberries
octan-1-ol	ethanoic acid	octyl ethanoate	oranges
amyl alcohol	ethanoic acid	amyl ethanoate	bananas
methanol	salicylic acid	methyl salicylate	oil of wintergreen
ethanol	methanoic acid	ethyl methanoate	rum (?)
iso-amyl alcohol	butanoic acid	iso-amyl butanoate	cherries

NB Other combinations could also be tried if you have time.
* At the conclusion of the experiment place all ester residues in the container in the fume-cupboard labelled "ORGANIC WASTES"

CONCLUSION:
Give a brief summary of your findings and write equations for any two reactions above.

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CHEMISTRY (LEVEL 4C)
EXPERIMENT 42.
PREPARATION OF PERSPEX

AIM:
To prepare poly(methyl methacrylate) "perspex" by the action of an initiating catalyst such as methyl ethyl ketone peroxide (MEKP). This is an example of **ADDITION** polymerization and the polymer formed is described as an "acrylic" polymer.

PROCEDURE:
Perform this experiment in the fume cupboard and wear disposable gloves as the monomer is toxic and malodorous.
(i) Obtain a moulding cup and ensure that the internal surface is clean and free from any moisture.
(ii) Approximately half fill the moulding cup with the perspex monomer and then add 10 drops of MEKP using the small plastic pipette provided.
(iii) Carefully mix in the MEKP catalyst using an icy-pole stick as a stirring rod. The polymerization reaction will now begin but there won't be any obvious changes for 15-20 minutes.
(iv) If you wish to embed an object in the perspex, then place this object carefully in the monomer in the moulding cup remembering that the *upper* surface of the object should be *downwards* in the moulding cup.
(v) Use the icy pole stick to manoeuvre the object to the correct position and to ensure all air bubbles are removed.
(vi) Now add more monomer to the moulding cup so that the cup is nearly full. Add the MEKP catalyst as before and stir carefully to prevent movement of the object being embedded.
(vii) Label your moulding cup and set the system aside for 24–48 hours.
(viii) When the polymer has totally hardened (i.e. polmerization is complete) the solid polymer unit may be pushed out of the moulding cup and retained.
(ix) Return the moulding cup to the trolley for further usage.

THEORY:
The monomer is methyl methacrylate which undergoes addition polymerisation as follows:

In this form of polymerization, the monomer "A" contains a double bond and the polymer forms by the double bond being changed to a single bond making two electrons available for chain propagation.
The polymer thus formed is :   ......A-A-A-A-A-A-A-A-A-A-A-A-A-A-A-A-..................
Because polymerization is dependent upon the monomer being unsaturated, it is called addition polymerization.

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CHEMISTRY (LEVEL 4C)
EXPERIMENT 43.
PREPARATION OF NYLON 6-10

AIM:
To react two immiscible liquids which form the condensation polmer "nylon 6-10" at the liquid-liquid interface.

PREPARATION:
The two solutions required have already been prepared for you. They are:

SOLUTION A = 5% (approx. 0.5 mol L^{-1}) aqueous 1,6-diaminohexane $H_2N(CH_2)_6NH_2$ solution made by dissolving 6.0 g of 1,6-diaminohexane and 2.0 g of NaOH pellets in 100 mL of distilled water. The solid 1,6-diaminohexane has a melting point of 39°C and needs to be melted by standing the reagent bottle in hot water for a few minutes.

SOLUTION B = 5% (approx. 0.2 mol L^{-1}) sebacoyl chloride $ClCO(CH_2)_8COCl$ solution made by dissolving 5 mL of sebacoyl chloride in 100 mL of hexane *or* trichloroethane.

PROCEDURE: * Avoid skin contact with either solution A or B!
(i) Wearing gloves, place 10 mL of the denser solution (solution B if trichloroethane was used as the solvent) in a clean 50 mL beaker. Use the approximate markings on the side of the beaker to gauge the 10 mL volume.
(ii) Now **slowly** pour 10 mL of the other reactant (the lower density one) down the side of the beaker held on an angle of about 45°. This should result in the second liquid floating on top of the denser liquid. Take care to minimize mixing at the interface.
(iii) Using a pair of tweezers, grasp the solid that forms at the interface and pull out the thread slowly whilst winding it onto a glass rod.
(iv) Wash the nylon fibre by dipping the fibre covered glass rod into a beaker of water.
(v) Leave the nylon to dry on the glass rod before removing it.
(vi) Any remaining reactants should be mixed thoroughly to produce nylon and then washed before discarding in a solid waste container in the fume cupboard.

THEORY:
Nylon is condensation polymer described as a "polyamide" being based upon peptide linkages;
i.e.

The name nylon 6-10 suggests that the polymer chain comprises linkages between a six carbon monomer A (1,6-diaminohexane $H_2N(CH_2)_6NH_2$) and an eight carbon monomer B (sebacoyl chloride $ClCO(CH_2)_8COCl$)
When these two monomers A and B polymerise to form nylon the small molecule HCl is eliminated.
The polymer formed is of the form: .... -A-B-A-B-A-B-A-B-A-B-...........   with the polymer chain linked by peptide bonds between each A and B.

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CHEMISTRY (LEVEL 4C)
EXPERIMENT 44 - A GRAVIMETRIC ANALYSIS FORMULA FOR HYDRATED MAGNESIUM SULFATE

AIM:

To determine the chemical formula for hydrated magnesium sulfate (Epsom Salts)

PREAMBLE:

Hydrated magnesium sulfate has the chemical formula $MgSO_4.xH_2O$ where x is an integer (whole number). With the careful heating of the solid crystals of $MgSO_4.xH_2O$, the water of crystallisation can be driven off leaving just crystals of the anhydrous compound $MgSO_4$.

PROCEDURE:

(i) Using a mortar and pestle, grind about 5 grams of the Epsom Salts crytals until they are now in the form of a powder rather than larger crystals.

(ii) Using a centigram electronic balance, determine the mass of a clean, dry crucible. Record this mass.

(iii) To the crucible add between 3 & 4 g of the $MgSO_4.xH_2O$ (Epsom Salts) crystals and determine the mass of crystals to the nearest 0.01 g. Record the mass of hydrated magnesium sulfate.

(iv) Using a cement mat, tripod and pipe-clay triangle, support the crucible and its contents so that they can be heated with a bunsen burner.

(v) Commence heating the crystals very slowly and gently ensuring that there is no loss of mass resulting from crystals being ejected from the crucible. (A crucible lid may assist in this situation if one is available.)

(vi) Continue heating and at the same time slowly increase the rate of heating until the crucible and remaining contents are approaching red-hot! As well as heating from beneath the crucible, heat the solid from above by carefully directing the bunsen flame into the top of the crucible.

(vii) After approximately 10 minutes of heating turn the bunsen burner off and allow the system to start cooling. Do not touch any of the apparatus as severe burns are likely.

(viii) Allow the crucible and contents to cool for about 10 minutes during which time, if available, the crucible and contents should be stored in a desiccator. Use crucible tongs to hold and move the crucible. The desiccator prevents the crystals re-absorbing moisture from the air.

(ix) When nearly back to room temperature, remove the crucible from the desiccator and without delay, determine the mass of crucible plus contents. Record this mass.

(x) Having finished, return the final crystals to the container labelled "$MgSO_4$" on the trolley.

CHEMICAL REACTION OCCURRING:

$$MgSO_4.xH_2O_{(s)} \rightarrow 1MgSO_{4(s)} + xH_2O_{(g)}$$

CALCULATIONS:

(i) Determine the mass of hydrated magnesium sulfate ($MgSO_4.xH_2O$) weighed out initially.

(ii) Determine the mass of anhydrous magnesium sulfate ($MgSO_4$) in the crucible finally. (**HINT**: If you've heated your crystals sufficiently, the mass you have now will be approximately *half* of the mass initially!)

(iii) By comparing your answers to (i) and (ii) above, determine the mass of water in the form of steam that was driven off during the heating process.

(iv) Given $M(MgSO_4)$ = 120.4 g mol^{-1} and using your answer to part (ii) above, calculate the number of mole of $MgSO_4$ formed i.e. calculate $n(MgSO_4)$ ....*

(v) Given $M(H_2O)$ = 18.02 g mol^{-1} and using your answer to part (iii) above calculate the number of mole of H_2O driven off i.e. calculate $n(H_2O)$ ....**

(vi) By comparing * and ** determine the value of the ratio $n(MgSO_4) : n(H_2O)$. In accordance with the chemical equation above, this ratio should be **1 : x** where **x** is an integer.

DISCUSSION:

(a) Epsom Salts are magnesium sulfate heptahydrate i.e. $MgSO_4.7H_2O$

(b) Discuss the sources of error in this experiment.

(c) What assumptions were made in this experiment?

(d) How might the procedure be improved so as to increase the accuracy?

CONCLUSION:

Give a brief (2 - 3 line) summary of the results obtained.

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