



Ideas for practical work

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INTRODUCTION

The Course Companion is written to help students but many teachers also find it useful. This section of the CD is for those who would like some ideas and actual details of practical experiments. It contains a list of possible experiments that could be performed for each core/AHL topic and for each option. It also gives practical details of 24 individual experiments. These are written for HL students but most of them, either as they are, or with minor changes, are also suitable for SL students.

Before using any of the following information it is essential that you read **Chapter 12 External and Internal Assessment**. From this chapter you will see that one of the real strengths of the IB programme is that unlike many national systems, there are no set experiments that must be done. All teachers are free to plan their own practical programme. This means that the ideas for experiments given here are only examples – many teachers, in many schools, will have many different experiments that they include as part of their practical programme. Much of the practical work does not have to be assessed and this again gives teachers and students more freedom of choice. Whether, or not, an experiment is formally assessed for the IB is a decision for the teacher.

The list of examples for each topic and option is just a list of headings. The way in which the teacher presents the information for an experiment will determine whether it is suitable for assessing Design (D), Data collection and processing (DCP) and/or Conclusion and evaluation (CE).

Apart from those mentioned in the book no further information about design experiments is given

here, as to do so would be to undermine the whole idea of a design experiment where students have to come up with their own research question and a detailed plan.

One of the main reasons for practical work is for students to understand the underlying chemistry. For the experiments, where the practical details have been given, questions and guidance are also included to help students to work out the answer. If a teacher decides to use one of these examples to DCP and/or CE, then the questions and guidance **must be deleted** as the students are not allowed this sort of help when being assessed. Many of the experiments given involve manipulation skills and as such are suitable for assessment of Manipulative skills (MS).

Remember, that important information about practical work and details of how to achieve high marks for the assessment are given in Chapter 12.

Whether, or not, it is to be assessed all experiments must address issues of the environment and safety. Different countries have different regulations regarding safety in the laboratory. It is vital that all experiments are checked by individual teachers to ensure they comply with national regulations before they are performed.

It is also sensible for each teacher and student to carry out their own risk assessment before attempting any experiment and the hazardous nature of any chemical used should be understood. In many cases small quantities are given to limit the effect on the environment and all waste material should be disposed of responsibly.

Possible laboratory investigations

Topic (Core/AHL) Investigation

1 Quantitative chemistry

- Determining the empirical formula of magnesium oxide.
- Investigating the factors influencing the solubility of salts.
- Determination of Avogadro's constant.
- Acid/base/carbonate titrations.
- Determination of the molar mass of an unknown acid.
- Analysis of over the counter aspirin tablets.
- Determination of the molar mass of an unknown gas/volatile liquid.
- Charles' law experiments to determine absolute zero.

2 and 12 Atomic structure

- Flame tests; emission spectra using a spectrometer.
- Investigating the principles behind a mass spectrometer.
- Plotting ionization graphs and other periodic trends.

3 and 13 Periodicity

- Physical and chemical properties of the elements Na–Ar.
- Reactions of the halogens and halide ions.
- Preparation and reactions of hydrogen halides.
- Properties of selected oxides and chlorides of Na–Ar.
- Oxidation states of Mn or V compounds.
- Complex ions of copper involving ligand exchange.

4 and 14 Bonding

- Physical and chemical properties of ionic/covalent compounds.
- Polarity of molecules using a microwave oven or burette and charged rod.
- Graphing of boiling points from data.
- Use of models/simulations for shapes of molecules/ions.
- Boiling points of liquid mixtures.

5 and 15 Energetics

- Determination of ΔH for reaction of zinc with $\text{Cu}^{2+}(\text{aq})$.
- Determination of enthalpies of neutralization.
- Investigate the energy content of liquid fuels.
- Indirect determination of ΔH values using Hess's Law.
- Design a hot or cold pack.

6 and 16 Kinetics

- Investigate the catalytic decomposition of hydrogen peroxide.
- Effect of particle size, temperature, concentration and addition of a catalyst on the rate of reaction.
- Investigate factors affecting the rate of a reaction of your choice.
- Determination of the rate law for the acid-catalyzed iodination of propanone.
- Determination of the activation energy for a reaction.

7 and 17 Equilibrium

- Determination of K_c for an esterification reaction.
- Reaction between $\text{Fe}^{3+}(\text{aq})$ and $\text{SCN}^{-}(\text{aq})$.
- Computer simulations for the Haber and Contact processes.

8 and 18 Acids and bases

- Investigate the action of buffers.
- Typical test tube reactions of acids and bases.

- Determination of the percentage of ethanoic acid in vinegar.
- Determination of the percentage of ammonia in window cleaner.
- pH of salts and common household items.
- pH titration curves for strong/weak acids and alkalis.
- Determination of pK_a for a weak acid.
- Determination of pK_{ind} for bromophenol blue.

9 and 19 Oxidation and reduction

- Investigate an aspect of an electrochemical cell.
- Simple electrolysis experiments.
- Determination of Faraday's constant.
- Determination of a reactivity series using redox reactions.
- How concentration affects a zinc/copper battery.
- Determination of an electrochemical series using cells.

10 and 20 Organic chemistry

- Simple test tube experiments to illustrate properties of different functional groups.
- Investigate the reactions of hydrocarbons.
- Use of models/simulations to show isomerism.
- Preparation of nylon 6,6.
- Oxidation reactions of primary, secondary and tertiary alcohols.
- Nucleophilic substitution reactions of halogenoalkanes.
- Investigate an aspect of esterification.
- Investigate an aspect of homologous series.

11 Uncertainty and error in measurements

- How accurate are the laboratory thermometers.
- Comparison of the equipment used to measure a stated quantity.

Option A: Modern analytical chemistry

- Visit to a modern analytical laboratory.
- Identification of substances by analysis of their spectra.
- Investigate the principles behind a mass spectrometer.
- Determination of concentration using colorimeter/visible spectrometer.
- Investigate the factors affecting retention time in chromatography.
- Separation of mixtures by chromatographic techniques.
- Investigate the efficiency of sunscreens.

Option B Human biochemistry

- Separation of amino acids using paper chromatography.
- Determination of ΔH_c using a food calorimeter.
- Biuret test for proteins.
- Determination of the degree of unsaturation in oils.
- Titrations with vitamin C tablets.
- Factors affecting the efficiency of enzymes.
- Use of models/computer simulations for nucleic acid structures.
- Making DNA fingerprints.

Option C Chemistry in industry and technology

- Visit to a local industrial plant.
- Separation of different metals from their ores.
- Determination of the percentage of copper in brass.
- Compute simulations for the Haber and Contact processes.
- Cracking of oil.
- Preparation of poly(phenylethene).

- Use of fuel cells.
- Computer modeling of nanotubes.

Option D Medicines and drugs

- Preparation and analysis of aspirin.
- Thin layer chromatography of mild analgesic tablets.
- Titrations with antacid tablets.
- Geometric/optical isomerism with models/computer simulations.
- Construct your own breathalyzer.
- Visit to a pharmaceutical company.
- Talk by a police officer/health worker on drug related problems.

Option E Environmental chemistry

- Determination of the sulfur content of a fossil fuel.
- Investigate an aspect of acid rain.
- Simple tests for water pollution.
- Determination of BOD using the Winkler method.
- Distillation of sea-water.
- Determination of the chlorine content of swimming pool water.
- Determination of the SO_2 content of polluted air.
- Determination of the NO_3^- and PO_4^{3-} content of polluted water.
- Visit to local sewage works.

Option F Food chemistry

- Determination of the degree of unsaturation of a vegetable oil.
- Analysis of foods past their sell by date to determine degree of rancidity.
- Determination of vitamin C content of a food.
- Analysis of the colour of food dyes using a visible spectrometer.
- The effect of pH on the colour of anthocyanins.
- Investigation of enantiomers with models/computer simulations.
- Compleximetric titrations with EDTA.

Option G Further organic chemistry

- Acid/base reactions of amines, phenols and carboxylic acids.
- Identification of specific aldehydes and ketones with 2,4-DNPH.
- Preparation of an alcohol using a Grignard reagent.
- A multi-stage synthesis e.g. 1,2-dibromocyclohexane from cyclohexanol via cyclohexane.
- Preparation of aspirin from ethanoic anhydride.
- Preparation of 1,3-dinitrobenzene from nitrobenzene.

INTRODUCTION: The aim of this practical is to observe and explain some important chemical reactions and to help non-native English speakers learn the English words for certain chemical terms.

ENVIRONMENTAL CARE: Use very small quantities of chemicals in small test tubes to minimise both cost and damage to the environment. Apart from the first two experiments all the rest involve 'heavy metal' ions, i.e. $\text{Cu}^{2+}(\text{aq})$, $\text{Ag}^{+}(\text{aq})$ and $\text{Ba}^{2+}(\text{aq})$. The residues remaining from these experiments must be poured into the waste container in the fume cupboard marked 'Heavy Metal Waste'. Do not pour heavy metal ions down the sink.

ASSESSMENT: As this practical is to enable you to familiarize yourself with the laboratory it will not be assessed formally.

REACTIONS: Carry out the following reactions. Observe carefully and test for any gases given off. Record all your observations directly into your practical book. Try to explain all the reactions taking place by writing balanced chemical equations.

- a. Add dilute hydrochloric acid to a small piece of magnesium.
- b. Carefully add a few drops of concentrated sulfuric acid to solid sodium chloride.
- c. Add dilute hydrochloric acid to solid copper(II) carbonate.
- d. In a dry test tube heat a little copper(II) carbonate. Using a test pipette, test for carbon dioxide with calcium hydroxide solution.
- e. To the residue obtained from reaction (d) add a little dilute hydrochloric acid and warm.
- f. Add silver nitrate solution to a solution of sodium chloride. Leave the product in sunlight for a few minutes.
- g. Add barium chloride solution to a solution of sodium sulfate.

SAFETY



When heating substances, and whenever concentrated acids are being used, you must wear safety glasses and a lab coat.

ENGLISH WORDS:

After you have completed the experiments you should make sure you are familiar with the following chemical terms:

- beaker, test-tube, measuring cylinder, analytical balance, pipette, burette.
- solute, solvent, solution.
- dilute, concentrated.
- mass, volume.
- acidic, basic, neutral, amphoteric.
- soluble, insoluble, precipitate.
- the names and chemical symbols for the first 32 elements in the Periodic Table.

INTRODUCTION: Neutralisation occurs between hydrogen ions and hydroxide ions in aqueous solution:



A suitable indicator is used, which changes colour at the 'end point' of the reaction when the pH of the solution corresponds to the presence of the appropriate salt and water only. In this experiment you will use the traditional method of titration to determine the number of moles of water of crystallisation in solid crystals of ethane-1, 2-dioic acid (oxalic acid) which has the formula $(\text{COOH})_2 \cdot x \text{H}_2\text{O}$. This traditional method makes use of volumetric flasks, pipettes and burettes and relies heavily on accurate readings of relatively large volumes.

ENVIRONMENTAL CARE: Although oxalic acid is quite poisonous it is a natural product (it occurs in the leaves of rhubarb) so dilute solutions of it should not affect the environment unduly and can be disposed of down the sink. At the end of this experiment you will carry out an environmental audit.

ASSESSMENT: This practical could be assessed formally for Data collection and processing (DCP), Conclusion and evaluation (CE) and Manipulative skills (MS). If used for formal assessment the following guidance and questions **must not be given to students**.

PROCEDURE: Weigh out accurately about 1.5 g of crystals of oxalic acid. Dissolve it in distilled water and make up the total volume to 250 cm³ in a volumetric flask. Pipette 25.0 cm³ of 0.100 mol dm⁻³ sodium hydroxide solution into a conical flask, add two drops of phenolphthalein and run oxalic acid solution from a burette into the flask, swirling continuously, until the indicator just loses its colour. Repeat the procedure to get two accurate results (within 0.10 cm³).

CALCULATION:

1. Write an equation for the reaction. Remember oxalic acid is a diprotic acid, i.e. one mole of acid reacts with two moles of sodium hydroxide.
2. What amount (in mol) of NaOH is present in 25.0 cm³ of 0.100 mol dm⁻³ sodium hydroxide solution?
3. What amount (in mol) of oxalic acid was present in the average volume required to react exactly with the sodium hydroxide solution?
4. What amount (in mol) of oxalic acid was present in your 250 cm³ volumetric flask?
5. What is the mass of one mole of oxalic acid?
6. How many molecules of water of crystallisation are present in one mole?

SAFETY



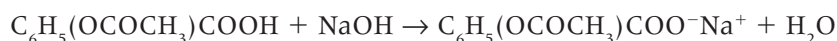
You must use a pipette filler and not your mouth whenever you use a pipette. Remember oxalic acid is poisonous.

DISCUSSION:

1. Estimate the degree of uncertainty in your readings using the balance, the volumetric flask, the pipette and the burette. How accurately can you quote your answer?
2. Compare your answer with the correct answer and work out the percentage error.
3. Suggest any other reasons for possible error.
4. Audit your experiment both financially and environmentally. Work out the total amounts of all reactants (ignore the few drops of phenolphthalein) and the total cost. What is thrown away during the experiment? Scale up your values for the whole class.

INTRODUCTION: For a long time the bark of the willow tree (*salix alba*) was used as a traditional medicine to relieve the fever symptoms of malaria. In the 1860's chemists showed that the active ingredient in willow bark is salicylic acid (2-hydroxybenzoic acid) and by 1870 salicylic acid was in wide use as a pain killer (analgesic) and fever depressant (antipyretic). However, because it is a relatively strong acid, salicylic acid has the undesirable side effect of irritating and damaging the mouth, esophagus and stomach membranes. In 1899 the Bayer Company of Germany introduced the ethanoate ester of salicylic acid, naming it 'Aspirin'. Since that time mild analgesics containing aspirin have appeared under many different brand names. The aim of this experiment is to determine the percentage of aspirin present in different commercial preparations and to find which is the best value for money.

The analysis makes use of the fact that aspirin is a monoprotic (monobasic) acid and therefore reacts with sodium hydroxide according to the equation:



ENVIRONMENTAL CARE: None of the reactants or products are particularly harmful to the environment and the waste can be safely disposed of down the sink.

ASSESSMENT: This practical could be assessed formally for Data collection and processing (DCP), Conclusion and evaluation (CE) and Manipulative skills (MS). If used for formal assessment the following guidance and questions **must not be given to students**.

PROCEDURE: Note the brand name and the price of the aspirin tablets you are using. Weigh out accurately one tablet (about 0.4–0.5 g) into a 50 cm³ conical flask and dissolve it in 10 cm³ of 95% alcohol. Titrate with 0.100 mol dm⁻³ sodium hydroxide solution using two drops of phenolphthalein solution as an indicator.

CALCULATIONS:

1. What amount (in mol) of sodium hydroxide was required to react exactly with the aspirin?
2. What amount (in mol) of aspirin was present in your weighed out tablet?
3. What is the mass of one mole of aspirin?
4. What is the percentage of aspirin in your sample?
5. Compare the mass of aspirin in the tablet that you have obtained with the value claimed by the manufacturer on the side of the box. What assumptions have you made that might not be true?
6. Pool your results with others and draw out a table for the different sources of aspirin showing their percentage purity and the cost per gram of pure aspirin. Which is the best buy?

SAFETY



No special precautions are necessary. You might like to consider why many doctors now recommend that you take paracetamol rather than aspirin for a headache even though aspirin is an effective mild analgesic.

4 Elements and compounds of the third period

INTRODUCTION: In going from left to right across the Periodic Table, the elements show a wide range of properties, changing from those typical of metals to those typical of non metals. This change in properties will be investigated by examining the elements of the second short period (Na-Ar) and some of their compounds. It will probably be easier if you present all your results and observations in tabular form.

ENVIRONMENTAL CARE: Use only very small quantities. Put all solid waste in the labelled beakers in the fume cupboard. Do not put solid waste down the sink.

ASSESSMENT: You should record your observations carefully and try to determine trends in behaviour but this practical will not be assessed formally.

PROCEDURE:

1. Prepare for the practical by drawing out a table using the data book to give the following information for each element;
 - the symbol
 - the electronic configuration
 - the atomic mass
 - the melting point
 - the boiling point.
2. *Physical appearance:* Record the physical appearance of the element, including its colour and whether it is a solid, liquid or gas at room temperature.
3. *Electrical conductivity:* By using a multimeter find out whether the element is a good or poor conductor of electricity. Try this out only on Mg ribbon, and the solid pieces of Al, Si and S.
4. *The action of air:* Heat pieces of Mg and Al directly in the flame. Heat S both in an ignition tube and on a spoon directly in the flame. Heat a very small amount of P in the fume cupboard. Wipe some Al foil with HgCl_2 solution and observe what happens.
5. *The action of acids:* For Mg, Al, Si and S mix a little of each element with a little dilute hydrochloric acid and observe. Identify any gases produced. If nothing happens, warm the tube gently.
6. *The action of alkalis:* For Mg, Al, Si and S mix a little of each element with a little dilute sodium hydroxide solution and observe. Identify any gases produced. If nothing happens, warm the tube gently.
7. *The chlorides:* Observe the physical form (solid liquid or gas) of NaCl, MgCl_2 , AlCl_3 and PCl_3 . Carefully add a little of each to some distilled water and observe what happens. Add a few drops of pH indicator to each solution.
8. *The oxides:* Mix a little of the oxides of Na (care), Mg, Al, Si, P (care) and S with distilled water. Note if any reaction occurs and then add a few drops of pH indicator to each solution.

SAFETY

You must wear safety glasses when heating the elements. The reactions of sodium will be demonstrated to you. Mercury(II) chloride solution is poisonous.

Questions



Metals and non metals behave differently. Comment on how this shows itself in experiments 3, 5, 6 and 8. Also comment upon any unexpected behaviour seen with Al and Si.

5 Chlorine content of swimming pool water

INTRODUCTION: It is important to use chlorine in swimming pools in order to stop the development of bacteria which would make the water both unhealthy and unsafe to swim in. The level must be carefully controlled. It must be high enough to inhibit bacterial growth, but low enough to stop skin and eye irritation. The normal safe level is around 1–3 ppm (parts per million). The following method for determining the chlorine content is very simple but gives surprisingly good results. Although you will be carrying out the procedure in the laboratory and understanding the chemistry involved it could easily be done by the side of any swimming pool by a non-specialist chemist.

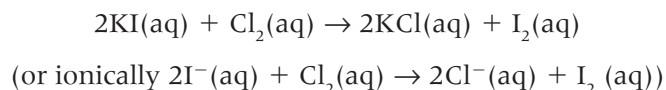
ENVIRONMENTAL CARE: This method uses very small quantities of relatively harmless substances and the waste can be disposed of down the sink.

ASSESSMENT: This practical could be assessed formally for Data collection and processing (DCP) and Conclusion and evaluation (CE). If used for formal assessment the following guidance and questions **must not be given to students**.

PROCEDURE:

1. Obtain fresh samples of the water from your local swimming pool (you might also like to determine the chlorine content of tap water).
2. Measure 300 cm³ of the water sample into a beaker, add 1 gram of solid potassium iodide and stir with a glass rod until the solid has dissolved. A brown colour of free iodine shows that chlorine is present in the sample.
3. Add three or four drops of starch solution which acts as an indicator for the titration by producing a blue/black colour in the presence of iodine.
4. Using a dropping pipette, add dropwise sodium thiosulfate (Na₂S₂O₃) solution with a concentration of 0.025 mol dm⁻³ making sure you count the drops. The colour of the solution should turn from black to green to blue and finally to colourless.
5. Stop when the blue colour just disappears and record the number of drops added. Now by an appropriate method determine the volume of each drop.

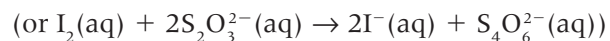
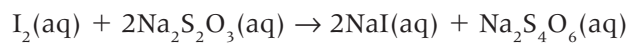
CALCULATION: An excess of potassium iodide is added so that all the available chlorine in the water is converted to iodine according to the redox equation:



SAFETY

As the method has been designed for use by non-chemists there are no special hazards to be aware of. You might like to think of the disadvantages, as well as the advantages, of adding chlorine to drinking water and suggest why ozone is now often used instead of chlorine by the water authorities.

The iodine produced reacts with the sodium thiosulfate by another redox reaction:



The amount of chlorine originally present can be determined from the amount of sodium thiosulfate used. Although the result could be given as a molarity, it is best to convert this into grams of chlorine per dm^3 of solution. If expressed as milligrams per dm^3 then this is the same as the value in parts per million. Is your swimming pool safe to swim in?

6

Boiling points of liquid mixtures

INTRODUCTION: This experiment aims to determine how the boiling point of a mixture of two fully miscible volatile solvents varies, as the composition of the mixture alters. Three different mixtures will be investigated and you will be required to interpret the results in terms of the intermolecular attractive forces between the component liquids. The three different mixtures are:

- $\text{CHCl}_3 / \text{CH}_3\text{COOC}_2\text{H}_5$ (trichloromethane/ethyl ethanoate)
- $\text{C}_2\text{H}_5\text{OH} / \text{C}_6\text{H}_{12}$ (ethanol/cyclohexane)
- $\text{C}_3\text{H}_7\text{OH} / \text{H}_3\text{CCH}(\text{OH})\text{CH}_3$ (propan-1-ol/propan-2-ol)

ENVIRONMENTAL CARE: The mixtures are all organic, so pour all the residues into the container marked 'Organic Waste' in the fume cupboard. Do not pour them down the sink. There is no need to wash out the apparatus afterwards with water as it can be left to dry by evaporation.

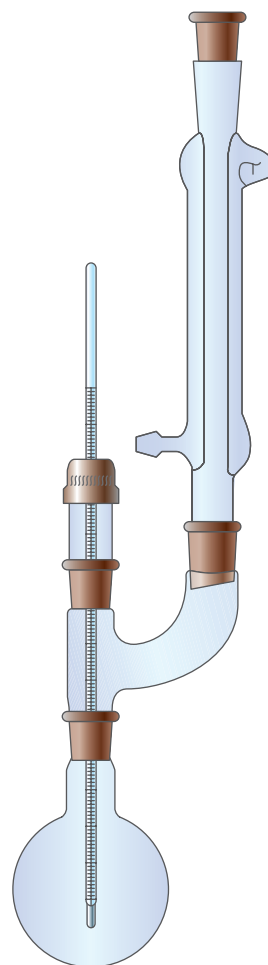
ASSESSMENT: This practical could be assessed formally for Data collection and processing (DCP), Conclusion and evaluation (CE) and Manipulative skills (MS). If used for formal assessment the following guidance and questions **must not be given to students**.

PROCEDURE:

- You will be allocated one of the three mixtures A, B or C. Carefully set up the reflux apparatus as illustrated.
- Turn the water on into the bottom of the condenser so there is a gentle flow out from the top into the sink and check the flow from time to time. The thermometer should dip into the liquid mixture, but not touch the walls of the flask. All the heating should be done with a small blue flame as the mixtures boil easily.
- Measure 10 cm^3 of one of the components directly from the burette into a 50 cm^3 round bottom flask and heat until a slow steady reflux is obtained. Record the temperature as the boiling point of the pure liquid.
- Measure 2 cm^3 of the second liquid into a clean dry test tube and, after moving the flame away and allowing the apparatus to cool briefly, add the second liquid by pouring it down the condenser.
- Reheat until a slow steady reflux is obtained and record the boiling point of the mixture. Take further readings after additions of 2 cm^3 portions, until a total of 10 cm^3 of the second liquid has been added.
- Allow to cool, dismantle the apparatus and pour the mixture away into the 'organic waste' container. Set up as before, and repeat the whole process starting with 10 cm^3 of the other solvent.
- Draw a single graph of boiling point against percentage composition by volume of the two components.
- Sketch the graphs obtained by others in the class for the other two mixtures.

SAFETY

The liquids used are all volatile and, apart from trichloromethane, are highly inflammable. Avoid breathing in their vapours as much as possible and keep them well away from a naked flame. This is particularly important when adding solvent to the reflux apparatus. The bunsen burner must first be moved to a safe distance. It is advisable to use a fume cupboard for the mixture containing trichloromethane.



Reflux apparatus

Questions



1. Compare the boiling points you obtained for the pure liquids with the values given in the Data Booklet. How accurate are your results?
2. What type of bonding exists between the molecules in the pure liquids? Does this help to explain their boiling points relative to their molecular mass?
3. What type of bonding exists between the molecules of the component liquids in the mixture? Is this similar or different to the pure liquids? How does this help to explain the shape of the graphs?

7 Enthalpy changes

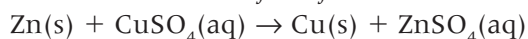
INTRODUCTION: The enthalpy changes for two different reactions will be determined practically. In the first experiment ΔH will be determined directly and account will be taken of heat losses by extrapolation of an appropriate graph. The second experiment involves the indirect determination of an enthalpy change using Hess's Law. The temperature can be measured manually but this experiment is ideal for using a data logger.

ENVIRONMENTAL CARE: The first experiment involves both copper and zinc and the residue should be placed in the container marked 'Heavy Metal Waste'. In the second experiment you will end up with two pure solutions of copper(II) sulfate. These should be placed in the marked bottle in the fume cupboard as they can be used for electrolysis experiments later.

ASSESSMENT: This practical could be formally assessed for Data collection and processing (DCP) and Conclusion and evaluation (CE). If used for formal assessment the following guidance and questions **must not be given to students**.

PROCEDURE:

1. Direct determination of ΔH for the reaction



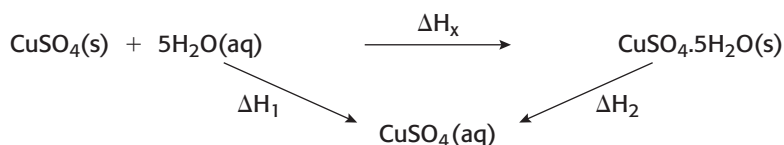
Using a measuring cylinder, put 50 cm³ of 0.20 mol dm⁻³ copper(II) sulfate solution into a polystyrene cup and record the temperature every half minute. After two minutes add 1.2 g of zinc powder and stir the mixture thoroughly and continuously, recording the temperature every thirty seconds. From the graph plotted record the predicted temperature rise if there had been no heat loss. Take the specific heat capacity of the solution as being equal to that of water, i.e. 4200 J kg⁻¹ K⁻¹, and ignore the heat capacity of the calorimeter, stirrer and thermometer/data logging probe to calculate the value for ΔH in kJ mol⁻¹.

2. Indirect determination of ΔH for the hydration of anhydrous copper(II) sulfate

If anhydrous copper(II) sulfate powder is left in the atmosphere it slowly absorbs water vapour giving the hydrated solid.



Heat is evolved in the reaction, but it is difficult to measure by the direct method. However it is possible to measure the heat changes directly when both anhydrous and hydrated copper(II) sulfate are separately dissolved in water and then construct an energy cycle to determine the required ΔH value indirectly.



SAFETY

There are no particular hazards associated with this practical. It is worth noting that both Cu²⁺ and Zn²⁺ are poisonous.

CALCULATIONS

1. Determine the enthalpy change ΔH_1 when 0.025 mol of anhydrous copper(II) sulfate is dissolved in 50 cm³ of water using the same method as before.
2. Determine the enthalpy change ΔH_2 when 0.025 mol of hydrated copper(II) sulfate is dissolved in 50 cm³ of water. (Note that 50 g water less 5×0.025 mole of water which comes from the hydrated salt should be weighed out).
3. Calculate ΔH_x from the values you obtain for ΔH_1 and ΔH_2 .

Redox titration with potassium permanganate (VII)

INTRODUCTION: An acidified solution of manganate(VII) or permanganate ions is a strong oxidizing agent and will be used in this practical to determine the percentage of iron in a sample of ammonium iron(II) sulfate.

The two relevant half-equations are:



and



ENVIRONMENTAL CARE: To save on chemicals this analysis has been scaled down and 10 cm³ pipettes and 100 cm³ volumetric flasks are used instead of the more normal 25 cm³ pipettes and 250 cm³ flasks. Manganese and iron, although essential minerals, are heavy metals and all residues containing them should be placed in the container in the fume cupboard marked 'Heavy Metal Waste'.

ASSESSMENT: This practical could be assessed formally for Data collection and processing (DCP), Conclusion and evaluation (CE) and Manipulative skills (MS). If used for formal assessment the following guidance and questions **must not be given to students**.

PROCEDURE:

1. Weigh out accurately about 2.5 grams of AR ammonium iron(II) sulfate crystals and dissolve them in about 40 cm³ of sulfuric acid with a concentration of approximately 1 mol dm⁻³. Do not heat the solution to assist dissolving.
2. Make up to 100 cm³ with distilled water in a volumetric flask and thoroughly mix the solution. Pipette 10 cm³ of this solution into a conical flask, add about an equal volume of distilled water and titrate with 0.0200 mol dm⁻³ potassium manganate(VII) solution to a faint pink colour.
3. Repeat the titration at least twice for accuracy, recording all your results.

CALCULATION:

1. Use the two half-equations to arrive at the overall equation for the reaction.
2. What amount (in mol) of Fe²⁺ are required to react with one mole of MnO₄⁻?
3. What amount (in mol) of MnO₄⁻ is present in your average titre value?
4. What amount (in mol) of Fe²⁺ is present in 10 cm³ of the Fe²⁺ solution?
5. What amount (in mol) of Fe²⁺ is present in 100 cm³ of the Fe²⁺ solution?
6. What mass of Fe²⁺ is present in the ammonium iron(II) sulfate you weighed out?

SAFETY



It is not advisable to get manganate(VII) ions on the skin as they stain. However, apart from the presence of dilute sulfuric acid there are no particular hazards associated with this practical.

7. What is the percentage of Fe^{2+} in ammonium iron(II) sulfate?
8. Find the correct chemical formula for ammonium iron(II) sulfate crystals from the bottle and calculate what the correct answer should be.
9. Compare your answer with the correct value and comment on your result.
10. Why do you think you should not heat the iron(II) salt to assist dissolving?

9 Group VII

INTRODUCTION: The elements fluorine, chlorine, bromine, iodine, and astatine make up Group VII of the Periodic Table. In this practical you will examine some of the properties and reactions of chlorine, bromine, and iodine and their ions.

ENVIRONMENTAL CARE: The elements are all oxidising agents and can potentially have an adverse effect on natural systems. Place all halogen residues in the marked container in the fume cupboard where any organic layer can be separated and the elements can eventually be converted into halides for disposal. Do not throw halogen residues down the sink. Halides, the salts of the halogens, all occur naturally in sea water and can be disposed of safely down the sink.

ASSESSMENT: This practical will not be assessed formally.

PROCEDURE:

1. Prepare for the practical by drawing out a table and using the data book give the following information for each element:
 - symbol
 - electronic configuration
 - atomic mass
 - melting point
 - boiling point
2. You will be shown pure samples of the three elements in the fume cupboard. Describe their physical appearance.
3. Solubility tests. Fill a micro test tube one quarter full with aqueous chlorine solution and add 10 drops of heptane. Stopper and shake well. Observe which layer appears to have the greatest concentration of chlorine. This may not be easy as chlorine solution only has a very pale green colour. Repeat this test with aqueous solutions of bromine and iodine, noting the colour in both layers.
4. Redox reactions. Line up four micro test tubes. In the first quarter fill with KCl(aq) , the second with KBr(aq) , the third with KI(aq) and the fourth with distilled water. To each tube add 10 drops of chlorine solution and observe the changes. A few drops of heptane and a good shake may help you to understand what is taking place. Wash out the tubes, refill with the same four solutions and now add bromine solution to each. Repeat a third time, adding iodine solution to each.
5. Test for halide ions. Line up three micro test tubes and quarter fill the first with KCl(aq) , the second with KBr(aq) and the third with KI(aq) . To each add 10 drops of silver nitrate solution and observe. Leave each solution in the light for 20 minutes and note any further changes.

SAFETY



All the halogens are poisonous (chlorine was used as a poison gas in the First World War). Use only small quantities, handle them with care, avoid inhaling the vapours and do not spill them on your skin or clothing. Fluorine is too dangerous for you to use.

Questions



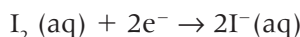
1. Which is the better solvent for the halogens and why?
2. Explain the chemistry behind the redox reactions giving the relevant equations.
3. Write an ionic equation for the reaction of chloride ions with silver nitrate solution.

Determination of the percentage of copper in brass

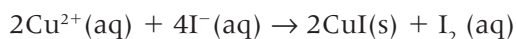
INTRODUCTION: The titration of iodine against thiosulfate is often used to determine the concentration of solutions of oxidizing agents. A known amount of the oxidizing agent is first added to an excess of an acidified solution of potassium iodide to liberate iodine. The freed iodine is then titrated against a standard solution of sodium thiosulfate. The relevant half-equations are:



and



As the thiosulfate reacts, the yellow colour of the iodine changes to a clear solution of iodide ions at the end point. However, it is difficult to see the precise point so starch is added as an indicator. The starch, which forms a deep blue coloured complex with iodine, should not be added until the solution is pale yellow, otherwise the iodine becomes strongly adsorbed on to the starch and the titration is less accurate. In this experiment a weighed sample of brass is dissolved in nitric acid to give a solution of copper(II) ions. Potassium iodide solution is then added and the copper(II) ions react to give a white precipitate of copper(I) iodide and liberate iodine which is then titrated against thiosulfate solution.



ENVIRONMENTAL CARE: Because potentially harmful copper ions (and other heavy metal ions) are involved, the experiment has been scaled down from normal. Transfer all residues containing material from the brass to the container marked 'Heavy Metal Waste'.

ASSESSMENT: This practical could be assessed formally for Data collection and processing (DCP), Conclusion and evaluation (CE) and Manipulative skills (MS). If used for formal assessment the following guidance and questions **must not be given to students**.

PROCEDURE:

1. Weigh accurately about 1.0 gram of brass and dissolve it in the minimum quantity of concentrated nitric acid in a 100 cm³ beaker.
2. Transfer the solution, with washings, to a 100 cm³ volumetric flask, add distilled water up to the mark and mix well.
3. Pipette 10 cm³ of the brass solution into a conical flask and add sodium carbonate solution until a slight permanent precipitate is obtained. This neutralises excess nitric acid in the solution.
4. Dissolve the precipitate in the minimum volume of dilute ethanoic acid and then add about 5 cm³ of approximately 1.0 mol dm⁻³ potassium iodide solution.
5. Titrate this solution against standard 0.100 mol dm⁻³ sodium thiosulphate solution.
6. Repeat the experiment to obtain consistent results.

SAFETY

Safety glasses must be worn when using concentrated nitric acid. The nitric acid must be added to the brass in the fume cupboard as poisonous nitrogen dioxide gas is evolved.

Questions



1. Give the equations for:
 - the reaction of the copper in the brass with concentrated nitric acid
 - the neutralisation of excess nitric acid with sodium carbonate
 - the overall reaction between iodine and thiosulfate ions.
2. Why is it necessary to neutralise the excess nitric acid and then re-acidify the solution with ethanoic acid ?

CALCULATION: Use your results to determine the percentage of copper in the sample of brass. Look up the composition of brass in a data book and compare your result.

INTRODUCTION: Ionic substances in the molten state, or in aqueous solution, conduct electricity. The mechanism of conduction involves the transport of ions and these ions are oxidised, or reduced, at their respective electrodes. The products that are evolved at the electrodes can depend upon a number of factors. The following qualitative experiments illustrate some of these factors.

ENVIRONMENTAL CARE: This practical involves five different electrolytic reactions. To minimise the use of chemicals move round each experiment in turn and use the same solutions or solid as the previous users. At the end of the practical the copper(II) sulfate solution can be filtered and returned to the marked bottle, and any lead salt remaining should be placed in the 'Heavy Metal Waste' container. The remaining solutions can be disposed of down the sink.

ASSESSMENT: This practical should be recorded and explained but will not be formally assessed.

PROCEDURE:

1. Using a suitable circuit with a potential difference of between 2 and 4 volts pass a current of between 0.1 and 1.0 amps through the following electrolytes.
2. Observe carefully all the changes taking place at the electrodes and also observe whether there is any obvious change to the electrolyte itself.
3. If gases are evolved try to identify them and note whether they are evolved at the positive or negative electrode. If gases are evolved at both electrodes observe whether they are given off in similar volumes.
 - *Copper(II) sulfate solution:* Using two copper electrodes allow the current to run for five minutes. Repeat the experiment using two graphite electrodes.
 - *Molten lead(II) bromide:* Connect the circuit whilst the lead(II) bromide is still solid. Heat gently in order to melt the solid and allow to run for a few minutes. Take care not to let the bunsen flame melt the connecting wires!
 - *Sodium chloride solution:* Using graphite electrodes pass the current through a dilute solution of sodium chloride for a few minutes then repeat the experiment using the more concentrated solution. After each experiment add a few drops of indicator solution to the electrolyte near each electrode.
 - *Potassium iodide solution:* Use graphite electrodes and allow to run for a few minutes.
 - *Water:* Try to electrolyse distilled water using graphite electrodes. If nothing happens add a few drops of dilute sulfuric acid to the water.

SAFETY



Take care when heating the lead(II) bromide. In some of the experiments small quantities of poisonous gases will be produced. Once you have identified these switch off the current.

Questions



1. Try to explain all your observations giving the relevant half-equations.
2. List the factors that can affect the discharge of ions during electrolysis and give one example of each from the above reactions.

12 Electrochemical cells

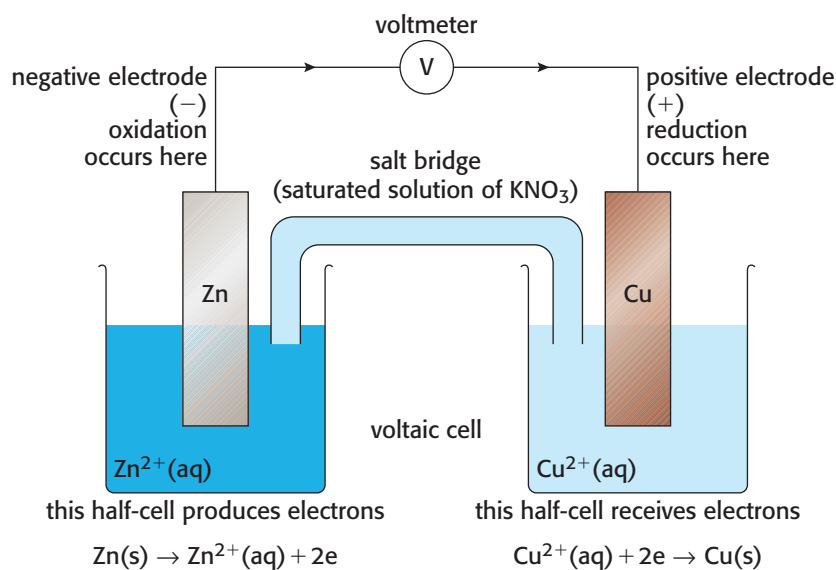
INTRODUCTION: This experiment demonstrates how a chemical reaction can be arranged to give out energy in the form of electricity and looks at some of the factors determining the value of E_{cell} .

ENVIRONMENTAL CARE: Do not pour solutions of heavy metal ions (Cu^{2+} , Fe^{2+} and Zn^{2+}) down the sink. Place all residues in the waste bottles provided in the fume cupboard.

ASSESSMENT: This practical will not be assessed formally.

PROCEDURE:

1. Construct a zinc/copper cell using 1.0 mol dm^{-3} solutions of Cu^{2+} and Zn^{2+} ions and strips of coppers and zinc with the two half cells connected by a salt bridge as shown in the diagram. Measure the cell e.m.f. using a high resistance voltmeter and try to determine which way the electrons are flowing in the external circuit.



2. From the relative tendencies of zinc, iron, and copper to lose electrons predict whether you would expect a zinc/iron cell to have a greater or smaller E_{cell} than the zinc/copper cell. Test your prediction by constructing a zinc/iron cell using the same zinc half cell connected to an iron half cell made with a piece of iron and a 1.0 mol dm^{-3} solution of Fe^{2+} . Assuming that each half-cell makes a fixed independent contribution to E_{cell} predict what the value of E_{cell} will be for an iron/copper cell under the same conditions. Test your prediction by constructing an iron/copper cell made from the two half cells used previously. How does your measured E_{cell} value compare with your predicted value?

SAFETY

This experiment does not involve any particular safety hazards.

3. The total e.m.f. produced by a cell depends on several factors, one of them is concentration. When current is drawn from a zinc / copper cell the reactions occurring at each electrode are:



However when the e.m.f. is being measured no current is being drawn and each electrode is at equilibrium.



Use Le Chatelier's Principle (the Equilibrium Law) to predict the changes which occur if the ion concentration in each equilibrium is reduced. What will be the effect of decreasing the concentration of copper ions on the tendency of the copper electrode to accept electrons from the zinc? What effect will this have on E_{cell} ? Test your prediction by setting up a zinc/copper cell using the zinc half cell previously used above but with the following concentrations of $\text{Cu}^{2+}(\text{aq})$, 0.100, and 0.001 mol dm^{-3} , making sure to rinse the salt bridge with distilled water each time.

Questions

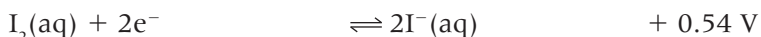
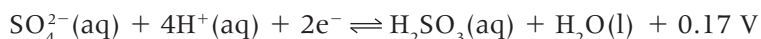
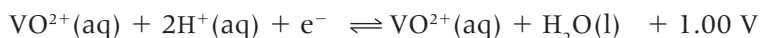
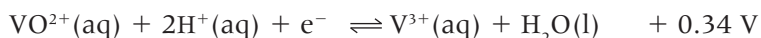
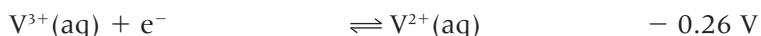
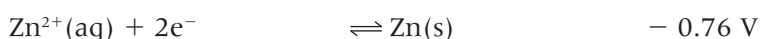


1. What are the standard conditions under which E_{cell} values are usually measured?
2. Using the E° values from the data book compare the values you obtained with the standard values.
3. What value might you expect for a cell made from your standard copper half cell connected to a zinc half cell where the concentration of zinc ions was 0.001 mol dm^{-3} ?
4. When the reaction between Zn(s) and $\text{Cu}^{2+}(\text{aq})$ reaches equilibrium what will E_{cell} equal?

INTRODUCTION: This experiment illustrates reactions involving different oxidation states of vanadium and asks you to explain them in terms of E° values for the relevant half equations. Given below are the colours of the different vanadium ions and some electrode potentials.

Hydrated ion	VO_2^+	VO^{2+}	V^{3+}	V^{2+}
Colour	yellow	blue	green	violet
Oxidation state	+5	+4	+3	+2

E°



ENVIRONMENTAL CARE: Solutions containing vanadium and zinc ions should be placed in the heavy metals container in the fume cupboard after use and not poured down the sink.

ASSESSMENT: This practical will not be assessed formally.

PROCEDURE:

- Preparation of standard solutions of vanadium(V) and vanadium(II):**
Place about 0.25 g of ammonium trioxovanadate(V) in a conical flask and add about 25 cm³ of dilute sulfuric acid. Carefully add about 5 cm³ of concentrated sulfuric acid and swirl the flask until you obtain a clear yellow solution. Pour about 2 cm³ of this vanadium(V) solution into each of two test tubes ready for later use. To the remainder in the conical flask add 1–2 g of zinc powder, a little at a time. Swirl the flask at intervals and record any changes taking place. When the solution has become violet (you may need to heat the flask for this final change) filter about 2 cm³ into each of three test tubes for your stock vanadium(II) solutions.
- Reactions of vanadium(V):** To one of the tubes containing vanadium(V) add about 2 cm³ of potassium iodide solution and mix. Then add about 2 cm³ of sodium thiosulfate solution. Record your observations. To the second of the tubes containing vanadium(V) add a little sodium sulfite and shake. Filter if cloudy. Now boil carefully in a fume cupboard to remove excess SO₂ and add one of the tubes of the vanadium(II) solution. Record all your observations.

SAFETY

Take care when adding the concentrated sulfuric acid. Safety glasses must be used and a lab coat must be worn. Do not add water to the concentrated acid.

3. *Reactions of vanadium(II)*: To the second of the tubes containing vanadium(II) add, a little at a time, an excess of potassium manganate(VII) solution, shaking after each addition, until no further change is observed. Using the electrode potentials given above predict a way in which you could oxidize vanadium(II) to vanadium(III) and no further. Use the remaining tube of vanadium(II) to test your prediction.

Questions



1. Try to explain the above reactions using relevant electrode potentials and by writing balanced overall equations.
2. Why was sodium thiosulfate added in one of the reactions?

Determination of K_c for an esterification reaction

INTRODUCTION: The equilibrium constant will be determined for the acid catalysed esterification of ethanoic acid with ethanol. The advantage of studying this particular reaction is that it is kinetically slow, so it is possible to measure the concentration of a reactant by titration without significantly disturbing the equilibrium in the short space of time it takes to carry out the titration.

ENVIRONMENTAL CARE: None of the reactants or products are damaging to the environment in the quantities used. Take care when handling the 'glacial' ethanoic acid.

ASSESSMENT: This practical could be assessed formally for Data collection and processing (DCP), Conclusion and evaluation (CE) and Manipulative skills (MS). If used for formal assessment the following guidance and questions **must not be given to students**.

PROCEDURE:

1. From the burettes provided make up four of the following mixtures. Run the liquids into the reagent bottles to give a total volume of 10 cm^3 then stopper immediately to prevent evaporation. Shake well then allow the bottle to stand at room temperature for a week to allow the mixture to reach equilibrium. Weigh separately 5 cm^3 of each of the liquids used to make up the mixtures (including the HCl) and record the mass.

Mixture	HCl/ cm^3	water/ cm^3	ethyl ethanoate/ cm^3	ethanoic acid/ cm^3	ethanol/ cm^3
1	5	0	5	0	0
2	5	1	4	0	0
3	5	2	3	0	0
4	5	3	2	0	0
5	5	0	4	0	1
6	5	0	4	1	0
7	5	0	0	1	4
8	5	0	0	2	3

2. After one week titrate the whole of the mixture in each bottle with 1.0 mol dm^{-3} sodium hydroxide using phenolphthalein as the indicator. In order to find the exact concentration of the HCl catalyst, also titrate approximately 3 mol dm^{-3} HCl with the 1.0 mol dm^{-3} sodium hydroxide solution. Record the results from another pair for the four mixtures you did not do.

CALCULATION: Give the balanced equation for the reaction between ethanoic acid and ethanol. For each mixture:

1. Calculate the amount (in mol) of ethyl ethanoate, ethanoic acid, ethanol, and water present in the original mixture, i.e before any reaction had taken place. When calculating the water amount remember to take into account the water present in the dilute HCl.

2. Calculate the amount (in mol) of ethanoic acid present in the equilibrium mixture and hence the amount (in mol) of ethanol, ethyl ethanoate, and water in the equilibrium mixture.
3. Calculate the equilibrium concentrations of the water, ethyl ethanoate, ethanoic acid, and ethanol in the mixture and hence the equilibrium constant.

Questions



1. Comment on the values obtained for the eight different mixtures.
2. ΔH for this reaction is $+ 17.5 \text{ kJ mol}^{-1}$. Would you expect the value of K_c to be greater or smaller at 60°C than at room temperature?
3. The yield of ester formed in the reaction increases considerably if concentrated sulfuric acid is added to the reactants. Suggest a reason for this.

INTRODUCTION: In this practical you will gain direct experience of using a pH meter and will plot a pH curve for a particular acid/base titration. From your own results and those of others in the group you will be asked to interpret the curves obtained for different acid/base pairs. This experiment provides a good opportunity to use a data logger with the pH probe.

ENVIRONMENTAL CARE: The products will be neutralised solutions of common salts which occur in sea-water and can be disposed of safely down the sink.

ASSESSMENT: This practical will not be assessed formally.

PROCEDURE:

1. Rinse the electrode with distilled water. Check the pH meter reading, making any necessary adjustments, by means of a standard buffer solution of pH 4 or 9, depending on the range likely to be used in the acid/base titration.
2. Pipette 25 cm³ of the chosen acid with a concentration of approximately 0.1 mol dm⁻³ into a tall 150 cm³ beaker.
3. Introduce the pH electrode and, if necessary, add distilled water until the electrode is properly immersed. Take a reading of the pH.
4. From a burette, run in standard 0.100 mol dm⁻³ sodium hydroxide solution in about 2 cm³ additions, recording the pH each time. About 5 cm³ from the approximate equivalence point, reduce the additions of alkali to 1 cm³ and finally to 0.1 cm³ nearer the end point.
5. Continue to add alkali and take the pH readings for a further 5 cm³. Plot the graph of pH against volume of alkali added either manually or using 'Excel' on the computer.

SAFETY



Although not strictly a safety point, be particularly careful when you handle the electrode as it is very fragile.

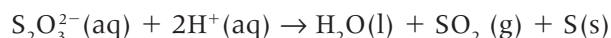
Questions



1. Make brief sketches of curves obtained by the rest of the class for other acid/base systems.
2. Explain the different curves in terms of whether the acid and bases involved are strong or weak.
3. For your particular experiment what is the accurate concentration (in mol dm⁻³) of the acid used?
4. If your experiment involved a weak acid, or weak base, what is the pK_a of the acid (i.e. NH₄⁺ for NH₃)?
5. What advantage/s (if any) has the pH meter over the use of an ordinary indicator?
6. What is meant by a 'buffer solution'? Give the composition of an example of a typical acidic buffer.

Factors affecting the rate of a chemical reaction

INTRODUCTION: Thiosulfate ions react in acid solution to produce a precipitate of sulfur according to the equation:



The rate of the reaction can be followed by measuring the time taken for a fixed amount of sulfur to be produced. This practical first examines the effect on the rate of the reaction when the concentration of thiosulfate ions is altered at a fixed temperature. Then it examines how the rate varies with temperature for a fixed concentration of thiosulfate ions.

ENVIRONMENTAL CARE: Sodium thiosulfate is known as photographers 'hypo' for fixing developed films and prints. To minimise pollution the concentrations of thiosulfate ions have been kept low and only very small amounts of sulfur dioxide are evolved. The residues can be disposed of down the sink.

ASSESSMENT: This practical could be assessed formally for Data collection and processing (DCP) and Conclusion and evaluation (CE). If used for formal assessment the following guidance and questions **must not be given to students**.

PROCEDURE:

1. **Concentration:** Measure 50 cm³ of 0.20 mol dm⁻³ sodium thiosulfate solution into a conical flask. Place the flask onto a white tile marked with a cross. Measure out 5 cm³ of 2.0 mol dm⁻³ hydrochloric acid into a measuring cylinder and then add the acid to the thiosulfate starting a stop-clock at the same time. Swirl the contents of the flask and then allow the flask to remain still on the tile. Look down vertically through the solution and time how long it takes for the cross to just disappear. Rinse the flask out immediately but take care not to get the cross on the tile wet. Then put 40 cm³ of the 0.20 mol dm⁻³ sodium thiosulfate solution into the flask and add 10 cm³ of water. Add 5 cm³ of the hydrochloric acid and time as before. Repeat the experiment four more times using 30, 20, 15 and 10 cm³ of the thiosulfate solution making the total volume up to 50 cm³ with water each time.
2. **Temperature:** Measure 50 cm³ of 0.040 mol dm⁻³ into the conical flask and warm until the temperature is one or two degrees above 20 °C. Place the flask on the tile marked with a cross and add 5 cm³ of 2 mol dm⁻³ hydrochloric acid, timing as before. Record the temperature of the mixture after the acid has been added. Repeat the experiment using fresh portions of the 0.040 mol dm⁻³ thiosulfate solution each time at temperatures of approximately 30, 40, 50 and 60 °C.

SAFETY



There are no particular hazards associated with this practical except that you should avoid breathing in any sulfur dioxide that is evolved while you are waiting for the cross to disappear.



Questions

1. Why do you think the flask should be rinsed immediately after each experiment?
2. Why is it important not to get the cross on the tile wet?
3. Using your results from experiment (1) plot a graph of molarity of the thiosulfate solution against time. Plot another graph of molarity against the reciprocal of time ($1/t$).
4. How does the rate of the reaction change with concentration? Suggest an explanation.
5. Using your results from experiment (2) plot a graph of time against temperature. Plot another graph of the reciprocal of time against temperature.
6. How does the rate of the reaction change with temperature? Suggest an explanation.
7. Apart from concentration and temperature state two other factors which can influence the rate of a chemical reaction.

The kinetics of the acid-catalysed iodination of propanone

INTRODUCTION: Propanone reacts with iodine in the presence of an acid catalyst according to the equation:



The rate of the reaction can be followed by measuring the time taken for the yellow colour of the iodine initially present to disappear. The rate equation can be expressed as:

$$\text{Rate} = k[\text{CH}_3\text{COCH}_3]^x [\text{I}_2]^y [\text{H}^+]^z$$

(where k = rate constant and x , y and z are the orders of the reactions with respect to propanone, iodine and hydrogen ions respectively). The aim of this practical is determine x , y and z and hence the overall order of the reaction. You will then be asked to propose a possible mechanism for the reaction which is consistent with the rate equation.

ENVIRONMENTAL CARE: Although propanone occurs naturally, halogenated hydrocarbons can pose particular problems and even though you are only dealing with dilute solutions transfer all the residues to the marked container in the fume cupboard. Do not dispose of them down the sink. The quantities used in this experiment have been reduced from those normally recommended to limit the effect on the environment.

ASSESSMENT: This practical could be assessed formally for Data collection and processing (DCP), Conclusion and evaluation (CE) and Manipulative skills (MS). If used for formal assessment the following guidance and questions **must not be given to students**.

PROCEDURE: For each experiment (a–g) use a burette to put the suggested volume of iodine solution into a test tube, and add the suggested volumes of acid and water from other burettes. Put the required amount of propanone into a separate tube. The two tubes should be added together, mixed quickly, and the stop-clock started. Note the time taken (in seconds) until the last trace of yellow colour disappears. At the end of the experiment pool your results with others in the class.

Reaction mixtures (all volumes in cm^3):

V_a = volume of 2.0 mol dm^{-3} propanone(aq)

V_b = volume of $0.0050 \text{ mol dm}^{-3}$ iodine(in $0.050 \text{ mol dm}^{-3}$ KI)

V_c = volume of 1.0 mol dm^{-3} sulfuric acid

V_d = volume of water

SAFETY



The product, iodopropanone, is a powerful lachrymator. Even the very small vapour pressure of it above the solution can make your eyes smart, unless you are careful to keep the tubes securely corked except when you are transferring samples.

Mixture	V_a	V_b	V_c	V_d	$[\text{CH}_3\text{COCH}_3]$ (m mol dm^{-3})	$[\text{I}_2]$ (m mol dm^{-3})	$[\text{H}^+]$ (m mol dm^{-3})
a	2	2	2	4	400	1.0	400
b	4	2	2	2	800	1.0	400
c	6	2	2	0	1200	1.0	400
d	2	1	2	5	400	0.5	400
e	2	0.5	2	5.5	400	0.25	400
f	2	2	4	2	400	1.0	800
g	2	2	6	0	400	1.0	1200

Questions



1. Calculate the values of x , y and z and hence give the overall rate equation.
2. Suggest a mechanism for the reaction which is consistent with the rate equation.

INTRODUCTION: The aim of this practical is to compare and contrast the chemical properties and reactivities of some simple organic molecules containing different functional groups.

ENVIRONMENTAL CARE: After you have carried out the following reactions place any residues remaining in the container in the fume cupboard marked 'Organic Waste'. Do not pour organic waste down the sink.

ASSESSMENT: This practical will not be formally assessed.

PROCEDURE:

1. *Solubility:* Generally only small polar molecules dissolve easily in water. Check this by adding a few drops of each of the following compounds to small quantities of cold water:
 - cyclohexane
 - ethanol
 - butan-1-ol
 - octan-1-ol
 - propanone
 - cyclohexanone
2. *Reactivity of the hydrocarbons:* Add a few drops of bromine solution to a few drops of each of the three hydrocarbons, cyclohexane, cyclohexene, and methylbenzene (used instead of benzene) in small test tubes. In the case of cyclohexane compare the effects of exposure to sunlight by placing one tube near the window and, to total darkness, by placing a second tube in the cupboard for the same length of time.
3. *Acidity and basicity:* Most organic compounds are neutral in their response to a pH test. The following compounds are different:
 - (a) Half fill four micro test tubes with distilled water. To the first add 4 drops of ethylamine solution, to the second 4 drops of phenylamine, to the third 4 drops of ethanoic acid, and to the fourth a small amount of benzoic acid. Add 4 drops of universal indicator to each tube, mix and determine the pH by the colour of the solution formed.
 - (b) To 4 drops of phenylamine, add 10 drops of distilled water. Does it dissolve? Now add 5 or more drops of dilute hydrochloric acid and observe whether there is any tendency to dissolve.
 - (c) To a small amount of benzoic acid, add 10 drops of distilled water. Does it dissolve? Now add 5 or more drops of dilute sodium hydroxide solution and observe what happens.

SAFETY



Organic compounds tend to be highly inflammable. Keep them away from naked flames and keep the stoppers on the reagent bottles when not in use. Use only small quantities and use a water bath for heating. As much as possible avoid breathing in their fumes.

4. *Two reactions of ethanol:*

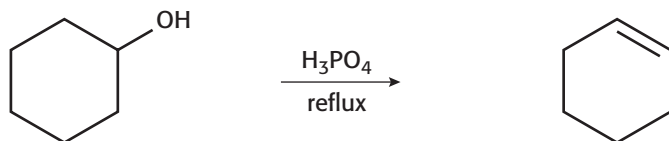
- (a) Oxidation. Mix 10 drops of potassium dichromate(VI) solution with 10 drops of dilute sulfuric acid. Add 4 drops of this mixture to 5 drops of ethanol in a micro test tube and warm in a beaker of hot water. Observe any changes in colour and smell.
- (b) Esterification. Mix 10 drops of ethanol with 10 drops of 'glacial' ethanoic acid and add 2 drops of concentrated sulfuric acid (care!). Warm the mixture in hot water for 5 minutes, then pour into 25 cm³ of dilute sodium carbonate solution. This removes the smell of any unreacted ethanoic acid. Smell the product carefully and try to describe it.

Questions

1. Try to explain your observation giving relevant equations for any reactions taking place.
2. Look up benzene on the chemistry HAZARD cards. Why was methylbenzene used instead of benzene in experiment 2? If benzene had been used how would you have expected it to react?

Preparation of cyclohexene from cyclohexanol

INTRODUCTION: In this experiment you will carry out the acid-catalysed dehydration of an alcohol to form an alkene according to the equation:



You will use the practical techniques of refluxing and separating two immiscible liquids using a separating funnel and use your results to calculate the percentage yield of the cyclohexene product you obtain.

ENVIRONMENTAL CARE: The aqueous waste can be safely disposed of down the sink, but the cyclohexene product must be placed in the container in the fume cupboard marked 'Organic Waste'. Do not pour cyclohexene down the sink.

ASSESSMENT: As this practical is for you to learn some practical techniques it will not be assessed formally.

PROCEDURE:

1. Carefully add 4 cm³ of phosphoric acid (85% strength) to a 50 cm³ round-bottom 'Quickfit' flask and add 10 cm³ of cyclohexanol making sure you know the exact mass of cyclohexanol added. Mix thoroughly by shaking and add a few anti-bumping granules.
2. Reflux the mixture using a small flame directly on the base of the flask for 15 minutes. Re-arrange the apparatus for distillation and distil very slowly collecting the distillate in a clean dry test tube.
3. Continue distilling until the residue in the flask becomes viscous and yellow. Pour the distillate into a separating funnel and add 2 cm³ of a saturated solution of sodium chloride. Shake the funnel and allow the two layers to separate.
4. Run off the lower aqueous layer and then run off the top layer into a small conical flask. Add a few pieces of anhydrous calcium chloride to the distillate, cork, and shake until the liquid is clear.
5. Decant into a previously weighed container and record the mass of product obtained. (N.B. The product at this stage will still be slightly impure and should really be distilled again, if time allows, with only the fraction collected between 80–85 °C being taken as pure.)
6. Using a few drops of your product devise a small test to show that the cyclohexanol has been converted into an unsaturated compound.

SAFETY



Cyclohexanol and cyclohexene are inflammable so keep them away from naked flames and as much as possible try to avoid breathing in their fumes. Phosphoric acid is a strong acid and safety glasses must be worn whilst the mixture is refluxing.

Questions



1. Calculate the percentage yield (assuming your product was pure cyclohexene).
2. Give the equation for the reaction you used to show that cyclohexene had been formed.
3. Why is it better to use phosphoric acid rather than sulfuric acid to dehydrate the alcohol?
4. What are the functions of:
(a) the saturated solution of sodium chloride
(b) the anhydrous calcium chloride?

INTRODUCTION: Nitrobenzene can be nitrated to 1,3-dinitrobenzene by heating with nitric acid in the presence of sulfuric acid. The product is obtained as fine pale yellow crystals. This experiment exemplifies the technique of purification by recrystallisation and illustrates the use of melting points and chromatography to determine the purity of the product.

ENVIRONMENTAL CARE: To minimise cost and the effect on the environment the procedure given below is for the small scale preparation of 1,3-dinitrobenzene. It involves considerably less chemicals than the traditional practical but does mean that care is required at all stages of the preparation to obtain a good yield. All organic waste should be disposed of in the appropriate container in the fume cupboard.

ASSESSMENT: This practical may be assessed for Manipulative skills (MS). If used for formal assessment the following guidance and questions **must not be given to students**.

PROCEDURE:

1. Using a small measuring cylinder and a teat pipette carefully transfer 1.5 cm^3 of fresh concentrated nitric acid and then 2 cm^3 of concentrated sulfuric acid to a 5 cm^3 round-bottom 'Quickfit' flask.
2. Place the flask on a top pan balance and using a new dropping pipette carefully add about 1.5 g of nitrobenzene and record the exact weight.
3. Fix an air condenser to the flask, swirl to ensure mixing of the reagents, and place in a beaker of boiling water. Reflux the mixture for 20 minutes swirling frequently then remove the water bath and allow to cool for five minutes.
4. Pour the contents of the flask into a beaker containing 25 cm^3 of tap water. Rinse out the residue of the flask with a little water if necessary. Allow the mixture to stand for five minutes and stir occasionally.
5. Filter the contents through a sintered glass crucible attached to a Buchner flask and water pump. Wash the solid product with a little water to remove the last traces of acid. Suck dry, and transfer the solid to a weighed watch glass.
6. Dry the product overnight and reweigh to determine the yield of crude 1,3- dinitrobenzene.
7. Meanwhile take a small quantity of the crude product and place in a 10 cm^3 test tube. Add no more than 1 cm^3 of ethanol and then warm the mixture in a hot water bath until you obtain a clear solution. Allow to cool.

SAFETY



Safety glasses must be worn when using concentrated acids. As much as possible avoid breathing in the fumes of nitrobenzene.

8. Filter the crystals obtained through the sintered glass crucible under reduced pressure. Wash with a little freezer-cooled ethanol and dry the crystals. Measure the melting point of the crystalline 1,3-dinitrobenzene and compare your value with the value given in the data book.
9. To test the purity of your product dissolve a few crystals in a few drops of trichloromethane. Place a spot of this solution 1 cm from the base on a microscope slide coated in silica. Place the slide in a jar containing a 50:50 mixture of trichloromethane and propanone as the eluent. Replace the lid on the jar and allow the eluent to rise up the slide. Mark the level reached by the eluent and then let the slide dry. Develop the chromatogram by placing the slide in another jar containing a few crystals of iodine.

Questions



1. Give the equation for the preparation of 1,3-dinitrobenzene and discuss the mechanism for the reaction.
2. State two ways in which the melting point of an impure substance may vary from that of the pure substance. What is meant by a 'mixed melting point determination'?
3. In the final part of the practical you have used tlc (thin layer chromatography). Write brief notes on other types of chromatography in particular column chromatography, paper chromatography and glc (gas-liquid chromatography). What is meant by the R_f value?

INTRODUCTION: Halogenoalkanes react with an aqueous solution of hydroxide ions according to the equation:



The following experiments examine the effect on the rate of hydrolysis when (a) the halogen is changed and (b) when primary, secondary and tertiary halogenoalkanes and an halogenoarene are used. Ethanol is used both as a good solvent and as a rather poor hydrolysis agent in place of hydroxide ions which tend to react too quickly for comparisons to be made.

ENVIRONMENTAL CARE: The aqueous residues will contain the heavy metal ions of Ag^+ and the organic residues will contain halogenated hydrocarbons so it is important not to dispose of any waste down the sink. Place all residues in the marked beaker in the fume cupboard. At the end of the practical the two immiscible layers in the combined waste can be separated and the aqueous layer transferred to the 'Heavy Metals' waste container and the remainder to the 'Organic Waste' container.

ASSESSMENT: This practical will not be formally assessed.

PROCEDURE:

- Experiment A:** Add a few drops of silver nitrate solution with a concentration of 0.05 mol dm^{-3} to separate aqueous solutions of NaCl, NaBr and NaI.
- Experiment B:** Repeat the above experiment by adding silver nitrate to a few drops of 1-bromobutane. Do not confuse a water/oil emulsion with a solid precipitate.
- This is a more quantitative experiment to compare the rates of hydrolysis. In each of three test tubes place 1 cm^3 of ethanol. Using separate test pipettes place 2 drops of 1-chlorobutane in the first test tube, 2 drops of 1-bromobutane in the second and 2 drops of 1-iodobutane in the third. Stand all three test tubes in a beaker of water at about $60\text{--}65^\circ\text{C}$ and place another test tube containing about 5 cm^3 of 0.05 mol dm^{-3} silver nitrate solution in the warm water. Wait until the contents of all the test tubes have reached approximately 60°C and then place 1 cm^3 of the silver nitrate solution into each of the other test tubes, and quickly shake each tube to mix the contents. Note and time carefully what you observe throughout the next five minutes.
- Repeat the above experiment using bromobenzene, 2-bromobutane and 2-bromo-2-methylpropane.

SAFETY

As only small quantities are being used there are no particular hazards associated with this practical. However, it is worth noting that some chlorinated organic compounds, e.g. dioxin, 2, 4, 5-trichlorophenoxyethanoic acid (Agent Orange) and polychlorinated biphenyls (PCB's) can be extremely poisonous.

Questions



1. What type of reagents (electrophiles or nucleophiles) react with halogenoalkanes? Explain your answer in terms of the polarity of the carbon-halogen bond.
2. Explain the significance of experiments (a) and (b).
3. Does the higher temperature have an effect on the reaction with 1-bromobutane? Explain.
4. Does changing the halogen atom affect the rate of hydrolysis? If so, explain why.
5. Place the different bromo-compounds in increasing order of their rates of hydrolysis and try to explain the order.

INTRODUCTION: These experiments illustrate some of the typical reactions of alcohols (ROH) and phenol ($\text{C}_6\text{H}_5\text{OH}$).

ENVIRONMENTAL CARE: Ethanol and methanol (wood alcohol) are natural products. They are completely miscible with water and small quantities can be safely disposed of down the sink. Ethanoic acid, which also occurs naturally as vinegar, can also be safely disposed down the sink. Phenol, however, is corrosive and could theoretically become chlorinated during water treatment so all phenolic waste must be disposed of in the container marked 'Organic Waste' in the fume cupboard.

ASSESSMENT: This practical will not be assessed formally.

PROCEDURE

Alcohols

1. Add a few drops of universal indicator to a few drops of ethanol in a test tube.
2. Add a small piece of sodium metal to about 1 cm^3 of ethanol in a test tube. When the reaction is complete and no sodium is left, evaporate to dryness, add a few drops of water and then test the pH of the solution.
3. Add a few drops of acidified potassium dichromate(VI) to about 1 cm^3 of ethanol and warm gently.
4. Add a few drops of ethanoic acid to a few drops of ethanol in a test tube. Add a few drops of concentrated sulfuric acid, warm gently then pour the mixture into a small beaker containing sodium carbonate solution.
5. Repeat step 4 using methanol instead of ethanol and a few crystals of salicylic acid instead of ethanoic acid.

Phenol

1. Add water to a crystal of phenol then a few drops of universal indicator.
2. Add a few drops of neutral iron(III) chloride solution to a crystal of phenol.

SAFETY



Alcohols are highly inflammable so keep them away from naked flames. Phenol is corrosive and must not come into contact with skin. You must wear safety glasses whenever you use phenol, sodium or concentrated sulfuric acid.



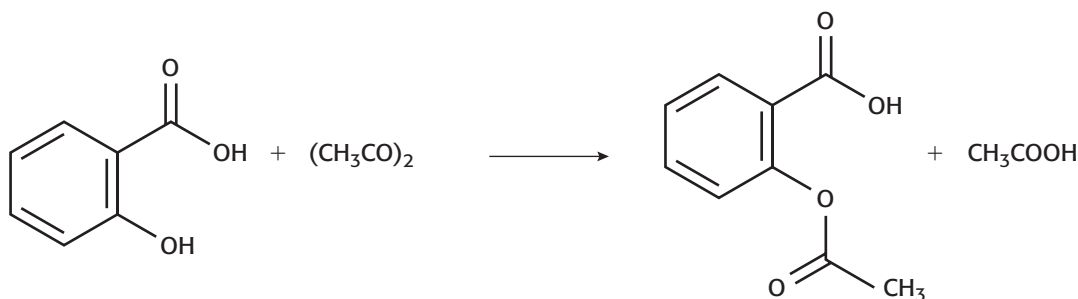
Questions

1. Compare and explain the relative acidities of water, ethanol and phenol. Which ion, out of hydroxide, phenoxide, and ethoxide, will be the strongest base?
2. Water is HOH and alcohols are ROH. In which of the above reactions is ethanol behaving in an analogous manner to water?
3. What class of compounds are formed when:
(a) primary alcohols are oxidised
(b) secondary alcohols are oxidised?

Would you expect tertiary alcohols to be readily oxidised?

4. Give a general equation for ester formation and give the specific equations for the esters formed in the reactions in steps 4 and 5. Why is the concentrated sulfuric acid added, and what is the function of the sodium carbonate solution in these experiments?
5. Explain why ethanol which has a relative molar mass of 46 boils at 78 °C whereas propane which has a relative molar mass of 44 boils at -42 °C.

INTRODUCTION: The aim of this experiment is to synthesise a pure sample of aspirin and test the product to see how pure it is. The preparation is relatively simple, but through the practical you will use the techniques of recrystallization and the use of the melting point apparatus. The synthesis is basically esterification, but instead of reacting an acid with the phenol group of the 2-hydroxybenzoic acid an acid anhydride is used in its place. The equation for the reaction is:



ENVIRONMENTAL CARE: The organic chemicals used are quickly broken in the environment and therefore present no threat.

ASSESSMENT: This practical may be assessed for Manipulative skills (MS). If used for formal assessment the following guidance and questions **must not be given to students**.

PROCEDURE

1. Shake together 2.0 g of 2-hydroxybenzoic acid with 4 cm³ of ethanoic anhydride in a 100 cm³ conical flask.
2. Now add 5 drops of concentrated sulfuric acid and continue agitating the flask. It may become quite warm for a while. Crystals of aspirin will appear and soon the mixture will form into a crystalline mass.
3. Now add 4 cm³ of cold 'glacial' ethanoic acid, and cool the flask in a beaker containing a mixture of crushed ice and water. Filter off the aspirin crystals onto a small filter, and wash once with ice water in order to remove residual acid.
4. Recrystallise the aspirin, by dissolving the crystals in a small quantity of hot water, and allow it to cool. A mass of very pure aspirin crystals should form. Filter them again, and leave overnight on a watch glass to dry completely.
5. Weigh the mass of pure dry aspirin and calculate the yield as a percentage of the theoretical maximum value. This may well be considerably below 100% because you have carried out a recrystallization, and have worked on a very small scale. Both lead to significant losses of material. In addition, many organic reactions either do not go to completion, or produce by-products.
6. Using the melting point apparatus determine the melting point of your sample and compare it with the Data Booklet value. If you have time you can further test the purity of your sample by a titration as described in *Experiment 3 Analysis of aspirin tablets*.

SAFETY

Ethanoic anhydride, 'glacial' ethanoic acid, and concentrated sulfuric acid are all corrosive. Treat them with care and wear an apron and safety goggles throughout the experiment.

INTRODUCTION: Two substances labelled MS and EB are provided and you are to choose one of them. A certain amount of analytical information is given for each and you should use this information to find out as much as you can about the structure of your compound. Strictly speaking this practical covers material from Option A, but all chemists should have some knowledge of the basics of spectroscopic techniques to determine structure.

You will then react your compound with sodium hydroxide solution following the procedure below. Two organic products are formed. One of these can be isolated and the other may be detectable by a chemical means but will not be isolated. Analytical information for these two compounds is also given. At the end of the experiment gather together all the information that you have collected and say as much as you can about the structures of the starting material and products that you have been associated with.

ENVIRONMENTAL CARE: The chemicals used present no serious risk to the environment.

ASSESSMENT: This practical will not be assessed formally.

PROCEDURE

1. Weigh 2.0 g of the substance (MS or EB) into a 50 cm³ round bottom flask, and add 10 cm³ of 2.0 mol dm⁻³ NaOH(aq) solution. Add a few anti-bump granules and gently reflux the mixture for 30 minutes. After cooling, pour the mixture into a small beaker and add dilute sulfuric acid solution, stirring until the solution is definitely acidic. A solid should form. Filter the solid using a Buchner flask and water pump. Wash the solid with a little distilled water. Keep both the solid and the aqueous filtrate.
2. Transfer the solid (call it MS-1 or EB-1) to a 50 cm³ conical flask, add about 20 cm³ of distilled water and bring to the boil. The solid should dissolve, although if some remains add a little more water until it does all dissolve. Set aside to cool slowly and a crop of good crystals should form. Filter these and transfer them to a previously weighed watchglass and leave to dry overnight in the air. Weigh again when the crystals are dry and then take the melting point of the pure dried product. Check the purity by thin layer chromatography using a silica plate with ethanol as the eluent. A UV lamp may be used to detect the spots.
3. The filtrate from the initial experiment contains another compound (call it MS-2 or EB-2) which will not be isolable but which may be detectable. Add 10 drops of potassium dichromate(VI) solution to the filtrate and warm gently. Observe any colour changes that occur.

SAFETY



Hot NaOH solution is damaging to the eyes. Safety glasses must be worn.

REPORT

Using the data gathered from your own experiments and the data provided below summarise this extensive experiment into a formal report. In your report record all the relevant information, and give as much information as you can upon the identities of all the compounds and on the reactions taking place.

Analytical information for MS and EB

Compound MS

Elemental analysis: 63.16% C, 5.26% H

Mass spectrum: molecular ion at 152 and fragments at 151, 137, 121, 93, 76, 59, 31 and 15.

IR peaks at 2900, 1700 (strong), 1650 and 1200 cm^{-1} .

^1H NMR: peaks at 3.8 (peak area 3 units), 7.0 (peak area 1 unit) and 7.3 ppm (peak area 4 units).

Compound EB

Elemental analysis: 72.00% C, 6.67% H

Mass spectrum: molecular ion at 150 and fragments at 121, 105, 77, 73, 45 and 29.

IR peaks at 2900, 1700 (strong), 1650 and 1200 cm^{-1} .

^1H NMR: peaks at 0.9 (peak area 3 units), 3.6 (peak area 2 units) and 7.3 ppm (peak area 5 units)

AN EXERCISE IN STRUCTURE DETERMINATION (CONT.)

Analytical information for MS-1 and EB-1

Compound MS-1

Elemental analysis: 60.87% C, 4.38% H

Mass spectrum: molecular ion at 138 and fragments at 137, 121, 93, 76, 45 and 17.

IR peaks at 3290 (broad), 2900, 2700, 1710 (strong), 1650 and 1200 cm^{-1} .

^1H NMR: peaks at 7.0 (peak area 1 unit), 7.3 (peak area 4 units) and 11.5 ppm (peak area 1 unit).

Compound EB-1

Elemental analysis: 68.85% C, 4.92% H

Mass spectrum: molecular ion at 122 and fragments at 121, 105, 77, 45 and 17.

IR peaks at 3290 (broad), 2900, 2700, 1710 (strong), 1650 and 1200 cm^{-1} .

^1H NMR: peaks at 7.3 (peak area 5 units) and 11.5 ppm (peak area 1 unit).

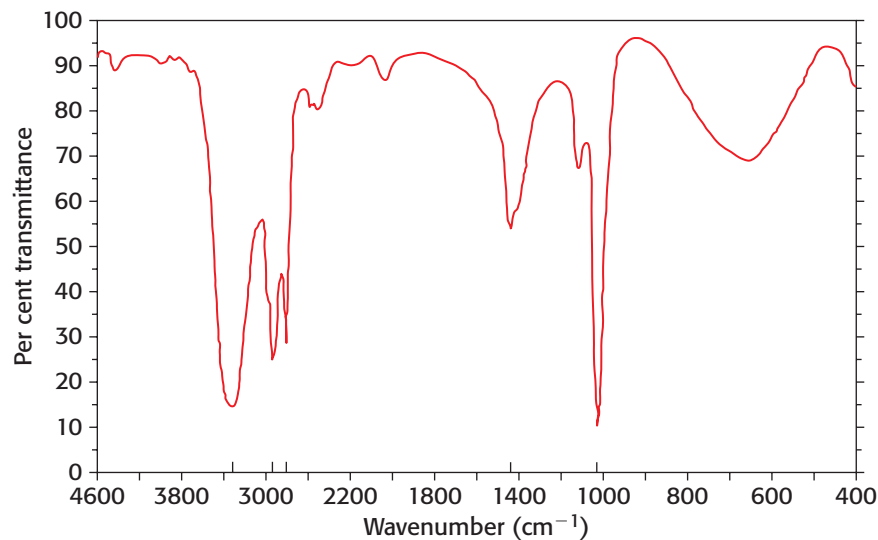
Analytical information for MS-2 and EB-2

Compound MS-2

Elemental analysis: 37.50% C, 12.50% H

Mass spectrum: molecular ion at 32 and fragments at 31, 17 and 15.

IR spectrum given below.



Infrared spectrum of compound MS-2

^1H NMR: peaks at 3.8 (peak area 3 units) and 4.5 ppm (peak area 1 unit).

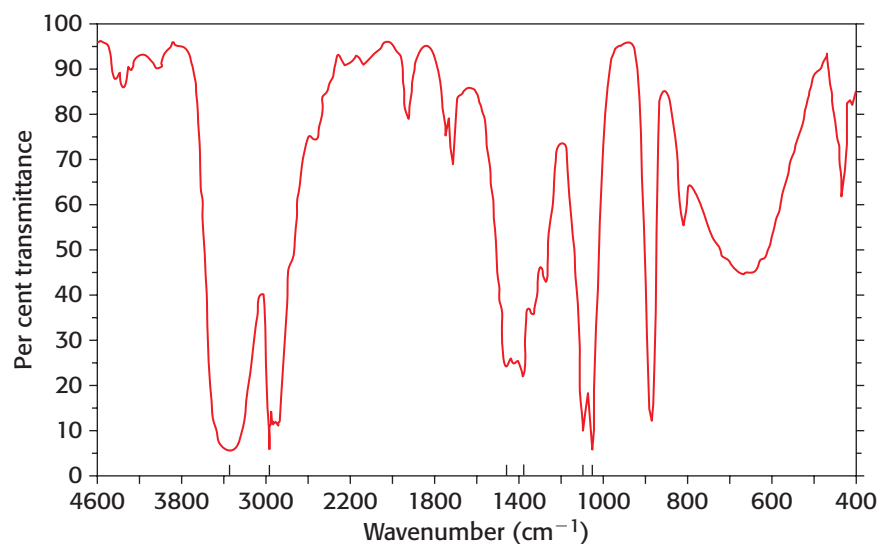
Compound EB-2

Elemental analysis: 52.17% C, 13.04% H

Mass spectrum: molecular ion at 46 and fragments at 45, 29 and 17.

IR spectrum given below.

^1H NMR: peaks at 0.9 (peak area 3 units), 3.6 (peak area 2 units) and 4.5 ppm (peak area 1 unit).



Infrared spectrum of compound EB-2