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Lab Book

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INTRODUCTION

Practical work is central to the study of chemistry. The International Advanced Subsidiary/Advanced Level (IAS/IAL) specification includes 16 core practical activities that link theoretical knowledge and understanding to practical scenarios. By completing the core practical activities, you will learn to:

- follow and interpret experimental instructions, covering a range of laboratory exercises throughout the course, with minimal help from your teacher
- manipulate apparatus, use chemicals, carry out all common laboratory procedures and use data logging (where appropriate)
- work sensibly and safely in the laboratory, paying due regard to health and safety requirements
- gain accurate and consistent results in quantitative exercises, and make the most of the expected observations in qualitative exercises.

By the end of this course, you should be able to use a variety of apparatus and techniques to:

- design and carry out both the core practical activities and your own investigations
- collect data that can be analysed
- use data to draw valid conclusions.

Your knowledge and understanding of practical skills and activities will be assessed in all examination papers.

- Papers 1 and 2 (IAS), and 4 and 5 (IAL) will include questions based on practical activities, including novel scenarios.
- Papers 3 (IAS) and 6 (IAL) will test your ability to plan practical work, including risk management and selection of apparatus.

Assessment for the Practical Skills Papers 3 and 6 will focus on four main areas:

- independent thinking
- use and application of scientific methods and practices
- numeracy and the application of mathematical concepts
- use of apparatus and equipment.

The ways in which you can demonstrate these practical skills are outlined in the tables on pages 4 and 5. You may wish to tick off each element as you gain confidence.

You will find answers and maths skills required for the practicals in the back of the book.

UNIT 1 (IAS): FORMULAE, EQUATIONS AND AMOUNT OF SUBSTANCE

1 Measurement of the molar volume of a gas

UNIT 2 (IAS): ENERGETICS

2 Determination of the enthalpy change of a reaction using Hess's Law

UNIT 2 (IAS): REDOX CHEMISTRY AND GROUPS 1, 2 AND 7

3 Finding the concentration of a solution of hydrochloric acid

4 Preparation of a standard solution from a solid acid and its use to find the concentration of a solution of sodium hydroxide

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5 Investigation of the rates of hydrolysis of some halogenoalkanes

6 Chlorination of 2-methylpropan-2-ol with concentrated hydrochloric acid

7 Oxidation of propan-1-ol to produce propanal and propanoic acid

8 Analysis of some inorganic and organic unknowns 1

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9b Investigating a 'clock reaction' (Harcourt–Esson, iodine clock)

10 Finding the activation energy of a reaction

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13a Redox titrations with iron(II) ions and potassium manganate(VII)

13b Redox titrations with sodium thiosulfate and iodine

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14 Preparation of a transition metal complex

UNIT 5 (IAL): ORGANIC NITROGEN COMPOUNDS: AMINES, AMIDES, AMINO ACIDS AND PROTEINS

15 Analysis of some inorganic and organic unknowns 2

UNIT 5 (IAL): ORGANIC SYNTHESIS

16 Preparation of aspirin

Practical skills	Core Practical							
Independent thinking in a practical context	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**
Solve problems set in a practical context								
Apply scientific knowledge to practical contexts								
Use and application of scientific methods and practices	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**
Identify and state how to control variables to improve experimental validity								
Present data in appropriate ways								
Evaluate results and draw conclusions								
Appreciate measurement uncertainties and errors								
Comment on the method for an experiment								
Numeracy and the application of mathematical concepts in a practical context	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**
Plot and interpret graphs								
Process and analyse data using appropriate mathematical skills								
Use appropriate numbers of significant figures based on the experimental data								
Consider the accuracy and precision of data								
Use of apparatus and equipment	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**
Recognise a range of laboratory apparatus and select appropriate apparatus for a particular scenario								
Understand how to use a range of apparatus and techniques appropriate to the knowledge and understanding included in the specification								
Consider the range and resolution of apparatus								
Identify health and safety issues and discuss how these may be dealt with								

Practical skills	Core Practical									
Independent thinking in a practical context	**9a**	**9b**	**10**	**11**	**12**	**13a**	**13b**	**14**	**15**	**16**
Solve problems set in a practical context										
Apply scientific knowledge to practical contexts										
Use and application of scientific methods and practices	**9a**	**9b**	**10**	**11**	**12**	**13a**	**13b**	**14**	**15**	**16**
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Consider the range and resolution of apparatus										
Identify health and safety issues and discuss how these may be dealt with										

Procedure

1. Set up the apparatus to capture and measure gas evolved from a reaction in the boiling tube.
2. Place 30 cm^3 of 1 mol dm^{-3} ethanoic acid in the boiling tube.
3. Place approximately 0.05 g of calcium carbonate in a test tube. Weigh the test tube and its contents accurately.
4. Remove the bung from the boiling tube and tip the calcium carbonate into the boiling tube. Quickly replace the bung in the boiling tube.
5. Once the reaction is over, measure the volume of gas produced.
6. Reweigh the test tube that contained the calcium carbonate.
7. Repeat the experiment six more times, increasing the mass of calcium carbonate by about 0.05 g each time. Do not exceed 0.40 g of calcium carbonate.

Learning tips

- Ensure that points plotted on a graph take up more than half the available space on each scale. Axes must occupy at least half of the space on the graph paper.
- Keep scales simple: one large square as 5 or 10 or 20 is ideal. A scale where one large square represents 3 or 7 units (or similar) is very difficult to plot on, and this often leads to errors.
- Always consider whether the graph line should go through the origin.
- Straight lines should be drawn with the aid of a ruler long enough to cover the full length of the line.

Objectives

- To find the volume of one mole of carbon dioxide gas

Equipment

- boiling tube
- stand and clamp
- bung fitted with delivery tube to fit boiling tube
- water bath for gas collection
- 100 cm^3 measuring cylinder
- 50 cm^3 measuring cylinder
- test tube
- mass balance (2 d.p.)
- 1 mol dm^{-3} ethanoic acid
- powdered calcium carbonate

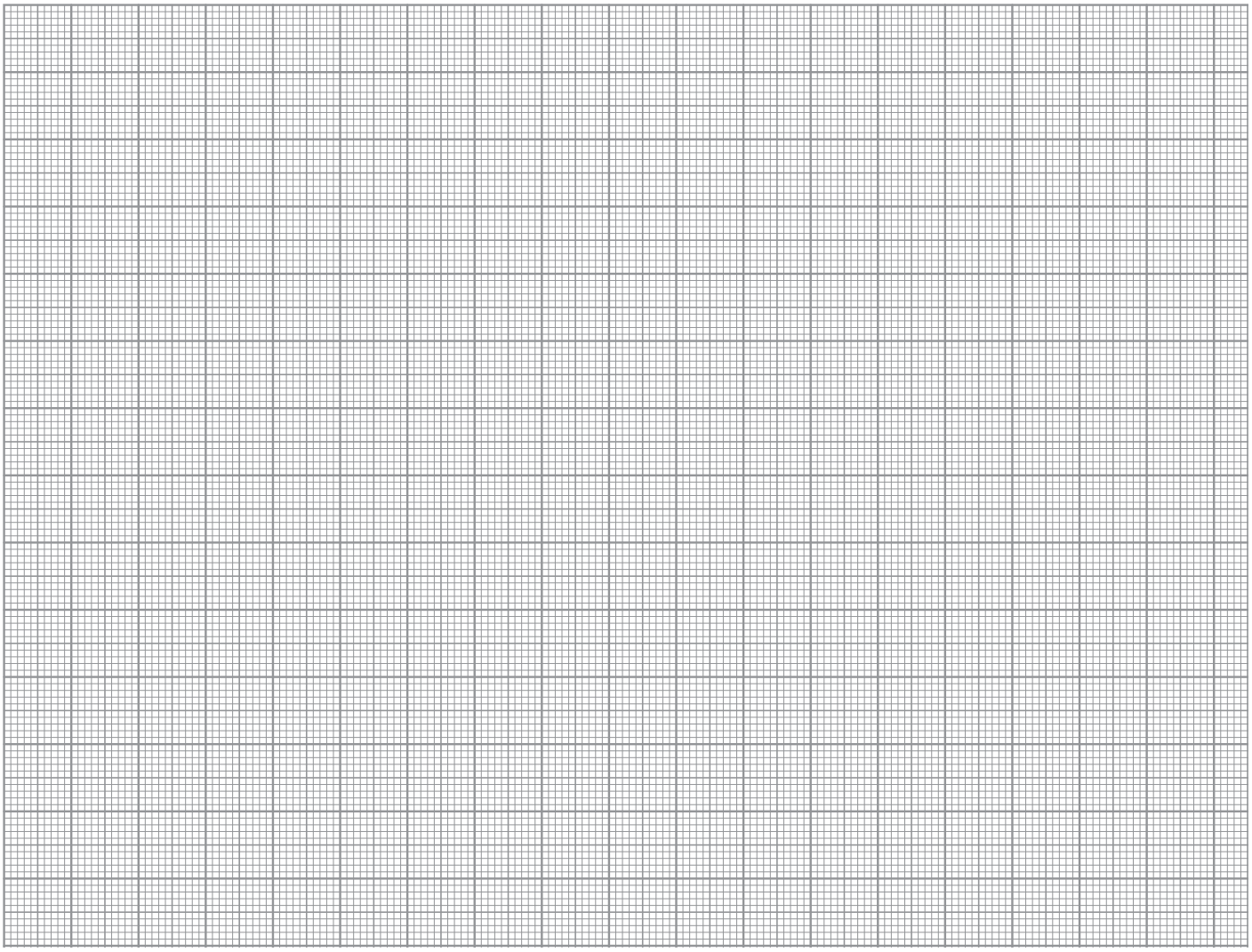
Safety

- Wear eye protection.
- Remove the bung if the delivery tube gets blocked, clear the blockage and repeat the procedure from the start.
- Avoid skin contact with the ethanoic acid, especially if the skin is broken or sensitive.

Results (Use this space to record your results.)

Analysis of results

1 Plot a graph of mass of calcium carbonate (on the *x*-axis) against volume of carbon dioxide collected (on the *y*-axis). Draw a straight line of best fit – this line must pass through the origin.



2 Use your graph to find the volume of carbon dioxide that would be made from 0.25 g of calcium carbonate.

3 In this reaction, one mole of calcium carbonate makes one mole of carbon dioxide. Calculate the number of moles of calcium carbonate in 0.25 g and hence calculate the volume of one mole of carbon dioxide gas in dm^3.

Questions

1 Write a chemical equation for the reaction between ethanoic acid, CH_3COOH, and calcium carbonate.

2 Why is it more accurate to find the mass of the calcium carbonate used by weighing the test tube containing the calcium carbonate and then reweighing the test tube after the calcium carbonate has been tipped out, rather than by weighing the empty test tube at the start?

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3 Identify the major source of error caused by the procedure used here.

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4 What change to the procedure/apparatus could you make to eradicate this error?

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5 Carry out two calculations to show that the ethanoic acid was in excess in all experimental runs.

Procedure

1. Place approximately 3 g of solid potassium carbonate in a test tube. Accurately weigh the test tube and its contents.
2. Use a burette to dispense 30 cm^3 of 2 mol dm^{-3} hydrochloric acid into a polystyrene cup, which is supported in a beaker.
3. Measure the temperature of the acid.
4. Gradually add potassium carbonate to the acid, stirring all the time and monitoring the temperature of the acid.
5. Reweigh the empty test tube.
6. Repeat steps 1–5 using approximately 3.5 g of potassium hydrogencarbonate instead of the potassium carbonate. This time, record the lowest temperature reached.

Results (Use this space to record your results.)

Mass of test tube with potassium carbonate/g	
Mass of test tube after emptying out potassium carbonate/g	
Mass of potassium carbonate used/g	
Start temperature/°C	
Highest temperature/°C	
Temperature change/°C	

Mass of test tube with potassium hydrogencarbonate/g	
Mass of test tube after emptying out potassium hydrogencarbonate/g	
Mass of potassium hydrogencarbonate used/g	
Start temperature/°C	
Lowest temperature/°C	
Temperature change/°C	

Objectives

- To calculate the molar enthalpy change for two reactions and use Hess's Law to determine the enthalpy change for the reactions

Equipment

- two test tubes
- 2 mol dm^{-3} dilute hydrochloric acid
- solid potassium carbonate
- solid potassium hydrogencarbonate
- thermometer able to read up to 50 °C or more
- polystyrene cup
- 250 cm^3 or 400 cm^3 beaker
- burette, clamp and stand
- stirring rod
- mass balance (2 d.p.)
- spatula

Safety

- Wear eye protection.
- Avoid skin contact with the reactants and products.

Learning tips

- You can assume that the heat capacity of the final solution is the same as the heat capacity of water. The volume of water produced in the reaction is so small it can be ignored.
- For exothermic reactions, the enthalpy change, $\triangle H$, is negative.
- Be careful to use equals signs correctly. It is very easy to end up stating that a negative number equals a positive number.

Analysis of results

1 The equations for the reactions occurring are:

reaction 1: $K_2CO_3(s) + 2HCl(aq) \rightarrow 2KCl(aq) + CO_2(g) + H_2O(l)$

reaction 2: $KHCO_3(s) + HCl(aq) \rightarrow KCl(aq) + CO_2(g) + H_2O(l)$

Calculate the energy change for each reaction in J. (The specific heat capacity of water is $4.2\,J\,g^{-1}\,°C^{-1}$.)

2 Calculate the enthalpy change, $\triangle H$, for each reaction in $kJ\,mol^{-1}$.
Assume that the hydrochloric acid is in excess.

3 Use your results to calculate the enthalpy change for the thermal decomposition of potassium hydrogencarbonate:

reaction 3: $2KHCO_3(s) \rightarrow K_2CO_3(s) + CO_2(g) + H_2O(l)$

Questions

1 Why is it not possible to measure the enthalpy change for the decomposition of potassium hydrogencarbonate directly?

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2 Show that the hydrochloric acid is in excess in both reactions.

3 Draw an energy level diagram for each reaction: 1, 2 and 3.

4 Explain why the reactions are conducted in a polystyrene cup rather than a glass beaker.

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Procedure

1. Wash out the 250 cm^3 volumetric flask with distilled water.
2. Use the pipette to transfer 25.0 cm^3 of the hydrochloric acid solution into the volumetric flask. Use distilled water to make the solution up to the mark.
3. Prepare your apparatus for the titration. The burette should contain the sodium hydroxide solution (previously standardised) and the conical flask should contain the dilute hydrochloric acid solution.
4. Pour a 25.0 cm^3 aliquot of the diluted hydrochloric acid into the conical flask. Use phenolphthalein as the indicator.
5. Titrate the contents of the flask against the sodium hydroxide solution. Record all burette readings to the nearest 0.05 cm^3.
6. The end point of this titration is indicated by the contents of the flask becoming pale pink. Continued swirling will cause the pink colour to fade and disappear. If the pink colour persists for 5 seconds or more, the end point has been reached.
7. Conduct further titrations until you have two concordant titres.
8. Ask your teacher or a technician to check one of your burette readings.
9. Record your results in the table below.

Learning tips

- Do not include your rough titration when calculating your mean.
- Give all burette readings to the nearest 0.05 cm^3.
- When you scale up to find the number of moles in the full volume of solution in the volumetric flask, use this equation:

$$\text{number of moles in full volume} = \frac{\text{full volume} \times \text{number of moles in aliquot volume}}{\text{aliquot volume in which you know the number of moles}}$$

Objectives

- To find the concentration of a solution of hydrochloric acid

Equipment

- burette, clamp and stand
- sodium hydroxide solution (approximately 0.08 dm^3, previously standardised)
- bench hydrochloric acid (approximately 1 mol dm^{-3})
- phenolphthalein
- 250 cm^3 conical flask
- 25 cm^3 volumetric pipette plus safety filler
- 100 cm^3 beakers for transfer of solutions
- funnel for filling burette
- 250 cm^3 beaker
- 250 cm^3 volumetric flask

Safety

- Wear eye protection. Goggles are preferred as sodium hydroxide is particularly hazardous to the eyes.
- Avoid skin contact with the acid, alkali and indicator.
- Always use a pipette filler; never use your mouth to suck the liquid up.
- Take care when clamping and filling the burette that it does not crack or topple over.

Results (Use this space to record your results.)

	Rough	Trial 1	Trial 2	Trial 3	Trial 4
Final burette reading/cm^3					
Initial burette reading/cm^3					
Titre/cm^3					
Concordant (Y/N)					

Analysis of results

1 Calculate the mean titre using your two concordant results.

2 Calculate the number of moles of sodium hydroxide that were contained in your mean titre.

3 Calculate the number of moles of hydrochloric acid that were contained in the full 250 cm^3 of diluted hydrochloric acid.

4 Calculate the concentration of the original solution of hydrochloric acid.

Questions

1 Write a chemical equation for the reaction of hydrochloric acid with sodium hydroxide.

2 The pink colour seen at the end point fades because the excess sodium hydroxide reacts with carbon dioxide in the air. This reaction produces sodium carbonate.

a Write an equation for the reaction of sodium hydroxide with carbon dioxide.

b When sodium hydroxide solution is stored, it reacts with carbon dioxide in the air.

i How will this change the concentration of the sodium hydroxide solution?

ii How will this affect the volume of sodium hydroxide solution required to reach the end point in the titration? Explain your answer.

3 Explain why it is better to have a titre of around 25 cm^3 than a titre of around 10 cm^3.

Procedure

1. Weigh an empty test tube. Scoop approximately 2.5 g of sulfamic acid into the test tube.
2. Accurately reweigh the test tube and its contents.
3. Dissolve the sulfamic acid in approximately 100 cm^3 of water in a beaker.
4. Transfer the solution, including the washings, to a 250 cm^3 volumetric flask and use distilled water to make up the solution to the mark.
5. Prepare your apparatus for the titration. The burette will contain the acid and the conical flask will contain the sodium hydroxide solution.
6. Pour a 25.0 cm^3 aliquot of sodium hydroxide solution of unknown concentration into the 250 cm^3 conical flask.
7. Use methyl orange as the indicator.
8. Titrate the contents of the flask against the sulfamic acid solution you prepared. Burette readings should be to the nearest 0.05 cm^3.
9. Conduct further titrations until you have two concordant titres.
10. Record your results in the table below.

Learning tips

- Indicators are very dilute weak acids. The more indicator you add, the less accurate your titration result will be.
- When conducting a titration, use distilled water to wash down the inside of the conical flask from time to time.
- When you scale up to find the number of moles in the full volume of solution in the volumetric flask, use this equation:

$$\text{number of moles in full volume} = \frac{\text{full volume} \times \text{number of moles in aliquot volume}}{\text{aliquot volume in which you know the number of moles}}$$

Objectives

- To make a solution of a known concentration of acid and use it to find the concentration of a solution of sodium hydroxide

Equipment

- burette, clamp and stand
- solid sulfamic acid
- sodium hydroxide solution of unknown concentration
- methyl orange indicator
- 250 cm^3 conical flask
- 25 cm^3 volumetric pipette plus safety filler
- 100 cm^3 beaker for transfer of solutions
- funnel for filling burette
- 250 cm^3 beaker
- 250 cm^3 volumetric flask
- mass balance (2 d.p.)

Safety

- Wear eye protection. Goggles are preferred as sodium hydroxide is particularly hazardous to the eyes.
- Avoid skin contact with sulfamic acid and sodium hydroxide.
- Take care when clamping and filling the burette that it does not crack or topple over.

Results (Use this space to record your results.)

	Mass of sulfamic acid = g				
	Rough	**Trial 1**	**Trial 2**	**Trial 3**	**Trial 4**
Final burette reading/cm^3					
Initial burette reading/cm^3					
Titre/cm^3					
Concordant (Y/N)					

Analysis of results

1 Calculate the concentration of your sulfamic acid solution. The M_r of sulfamic acid is 97.1.

2 Calculate the mean titre using your concordant results.

3 Calculate the number of moles of sulfamic acid in your mean titre.

4 Sulfamic acid is a monoprotic acid. This means that one mole of sulfamic acid will react exactly with one mole of sodium hydroxide. Calculate the concentration of the sodium hydroxide solution used.

Questions

1 A 250 cm^3 volumetric flask has an accuracy of ±0.6 cm^3. Calculate the percentage uncertainty in the volume of the sulfamic acid solution in the volumetric flask.

2 Each burette reading is accurate to ±0.05 cm^3. Calculate the percentage uncertainty in one of your titres.

3 Why should the pipette be rinsed with the sodium hydroxide solution after it has been washed with water?

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4 Why is there no need to dry the conical flask after washing it out between trials?

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5 Identify another indicator you could use in this titration, and state the colour change you would see at the end point.

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Procedure

Part 1

1. Set up a water bath by filling the 250 cm^3 beaker up to the three-quarters mark with water at around 50 °C.
2. Take three test tubes and add 5 cm^3 of ethanol to each one.
3. Add four drops of 1-iodobutane to the first tube, four drops of 1-bromobutane to the second tube and four drops of 1-chlorobutane to the third tube. Label the tubes.
4. Loosely place a bung in each test tube and place the test tubes in the water bath.
5. Take three clean test tubes and pour 5 cm^3 of silver nitrate solution into each one. Then place the test tubes in the water bath.
6. When the halogenoalkane–ethanol solutions have reached the temperature of the water bath, add one test tube of silver nitrate solution to one of the halogenoalkane–ethanol solutions and replace the bung. At the same time, start the stop clock.
7. Measure the time taken for a precipitate to appear. As soon as the solution becomes cloudy, stop the stop clock.
8. Repeat steps **6** and **7** for the other two halogenoalkanes.

Part 2

9. Repeat Part 1 using 1-bromobutane, 2-bromobutane and 2-bromo-2-methylpropane instead of the other halogenoalkanes.

Results (Use this space to record your results.)

Objectives

- To investigate the relative rates of hydrolysis of primary, secondary and tertiary halogenoalkanes and of chloro-, bromo- and iodoalkanes

Equipment

- 250 cm^3 beaker
- 12 test tubes with bungs
- 1-chlorobutane
- 1-bromobutane
- 1-iodobutane
- 2-bromobutane
- 2-bromo-2-methylpropane
- 0.05 mol dm^{-3} silver nitrate solution
- 15 cm^3 ethanol
- dropping pipettes
- two 10 cm^3 measuring cylinders
- stop clock
- labels for test tubes
- kettle

Safety

- Wear eye protection.
- Avoid skin contact with the reactants.
- There must be no naked flames in the vicinity as ethanol and halogenoalkanes are highly flammable.
- The laboratory needs to be well ventilated to prevent the inhalation of fumes.

Learning tips

- The hydrolysis of halogenoalkanes is a nucleophilic substitution reaction.
- In this investigation, the nucleophile is water.
- NaOH can be used instead of water to hydrolyse the halogenoalkanes but then any excess NaOH must be neutralised by HNO_3 before the $AgNO_3$ is added. Otherwise a precipitate of Ag_2O will form.

Analysis of results

1 Describe the pattern shown in your results for Part 1.

2 Describe the pattern shown in your results for Part 2.

Questions

1 Write an equation for the reaction of 1-bromobutane with water.

2 In these reactions, a precipitate forms. Identify the precipitate formed when the halogenoalkane is 1-iodobutane.

3 Explain why ethanol is used in these reactions.

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4 Explain why water is able to act as a nucleophile.

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5 Explain why water is used as the nucleophile rather than hydroxide ions.

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6 Draw skeletal formulae for each of the halogenoalkanes used in this investigation (there are five of them). Classify each halogenoalkane as primary, secondary or tertiary.

Procedure

1. Pour 10 cm^3 of 2-methylpropan-2-ol and 35 cm^3 of concentrated hydrochloric acid into a large conical flask. Very gently swirl the contents of the flask.
2. Place the bung in the mouth of the flask. Gently swirl again, then remove the bung to release the pressure.
3. Continue swirling the mixture with the bung fitted, and then releasing the pressure, for around 20 minutes. You should see two layers in the flask. The upper layer is the crude product.
4. Add approximately 6 g of powdered anhydrous calcium chloride to the flask and swirl until it has dissolved. This will ensure that any unreacted alcohol is in the lower aqueous layer.
5. Transfer the reaction mixture to a separating funnel. Allow the mixture to settle into the two layers. Run off and discard the lower layer. Retain the upper organic layer in the separating funnel.
6. Add approximately 20 cm^3 of sodium hydrogencarbonate solution to the separating funnel. Swirl the funnel. The production of carbon dioxide will cause the pressure to increase; remove the bung at frequent intervals to release this pressure. Run off and discard the lower aqueous layer.
7. Repeat the washing with sodium hydrogencarbonate solution, shake the separating funnel, and release the carbon dioxide gas produced, at frequent intervals.
8. Run off and discard the lower layer. Ensure none of the aqueous layer remains in the tap.
9. Run off the organic layer into a small conical flask. Add a full spatula of anhydrous sodium sulfate. Place the bung in the flask and swirl the contents to mix. Leave the mixture until the liquid looks completely clear, swirling occasionally.
10. Decant the organic liquid into a 50 cm^3 pear-shaped (or round-bottomed) flask.
11. Set up the flask for distillation.
12. Collect the fraction boiling between 50 °C and 52 °C.
13. Place your pure product in a labelled sample tube.
14. Carry out the test described in the 'Analysis' section of this practical.

Learning tips

- The OH group in an alcohol can be replaced by a halogen. As well as the method used here to chlorinate an alcohol, PCl_5 can be used to make a chloroalkane. HBr (which is made in situ) can be used to make a bromoalkane. Red phosphorus with iodine can be used to make an iodoalkane.
- You can check the purity of a substance by measuring its boiling temperature.

Objectives

- To produce and purify a sample of 2-chloro-2-methylpropane

Equipment

- 250 cm^3 conical flask with bung
- 100 cm^3 (or larger) separating funnel with bung
- 250 cm^3 beaker for liquid run off from separating funnel
- filter funnel to fit separating funnel
- apparatus for distillation with 50 cm^3 pear-shaped (or round-bottomed) flask and thermometer able to read up to 100 °C
- 25 cm^3 and 100 cm^3 measuring cylinders
- 2-methylpropan-2-ol
- 0.1 mol dm^{-3} sodium hydrogencarbonate solution
- 6 g powdered anhydrous calcium chloride
- anhydrous sodium sulfate
- small conical flasks with bungs
- sample tube
- 0.05 mol dm^{-3} silver nitrate solution
- dilute (0.5 mol dm^{-3}) sodium hydroxide solution
- dilute (0.1 mol dm^{-3}) nitric acid
- 70 cm^3 concentrated hydrochloric acid
- test tubes
- spatulas
- 5 cm^3 ethanol
- Bunsen burner
- beaker for water bath

⚠ Safety

- This procedure requires the use of a working fume cupboard.
- Wear eye protection. Goggles are preferred.
- Avoid skin contact with the reactants and products. Wear gloves.
- Avoid inhaling vapours.
- The product of the distillation process is flammable.
- The fumes from the concentrated hydrochloric acid are toxic and corrosive and must not be inhaled, especially by anyone with a respiratory problem.

Results (Use this space to record your results.)

Analysis of results

Perform the following test on the distillate.

1 Place a few drops of the distillate in a test tube.

1 Add 5 cm^3 of ethanol and 1 cm^3 of aqueous sodium hydroxide to the test tube.

3 Warm the mixture in a water bath.

4 Add excess nitric acid to the mixture followed by a few drops of silver nitrate solution.

Describe what you see.

Questions

1 Write an equation for the reaction of 2-methylpropan-2-ol with concentrated hydrochloric acid.

2 What is removed from the crude product when it is shaken with sodium hydrogencarbonate solution? Write an equation for any reaction that occurs.

3 2-methylpropan-2-ol has a boiling temperature of 82 °C and is soluble in water.
2-chloro-2-methylpropane has a boiling temperature of 51 °C and is insoluble in water.
Explain these differences.