

eGUIDE//

Centres are responsible for their own hazard analysis and risk assessment before beginning this practical work with pupils.

Chemistry

Unit A21: Practical Manual

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Practical 1.1

Investigating the effect of changing concentration on the rate of a chemical reaction (spec ref 4.3.7)

Introduction

An aqueous solution of sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$) reacts slowly with dilute hydrochloric acid to form an insoluble precipitate of sulfur.



The sulfur made by this reaction appears as a yellow solid precipitate. In this experiment the concentration of sodium thiosulfate will be varied by dilution and the time (in seconds) for a given mass of sulfur to form is recorded.

The rate of reaction is defined as the change of the concentration (amount) of a reactant or product with respect to time. The rate of this reaction can be monitored by measuring the time that it takes for a fixed amount of the sulfur precipitate to be produced.

As the reaction time is measured until the same, fixed amount of sulphur is produced, we can say that:

Rate = amount of sulfur/time or rate \propto 1/time

A conical flask containing the reaction mixture of sodium thiosulfate solution and hydrochloric acid is placed on top of a piece of card that has been marked with an **X**.

This rate of reaction is monitored by viewing the reaction in the conical flask and recording how long it takes for an **X** placed beneath it to “disappear”. The **X** is eventually obscured by the sulfur precipitate.

X still visible



X partially obscured



X disappeared





Practical 1.1

Investigating the effect of changing concentration on the rate of a chemical reaction (spec ref 4.3.7)

Apparatus and materials

- safety glasses
- stop clock
- 100 cm³ conical flask
- 2 × plastic dropping pipettes
- 3 × 10 cm³ measuring cylinder
- card marked with an X
- hydrochloric acid (1.0 mol dm⁻³)
- sodium thiosulfate solution (0.1 mol dm⁻³)
- bottle of deionised water

Procedure

1. Use a 10 cm³ measuring cylinder to measure out 10 cm³ of 0.1 mol dm⁻³ sodium thiosulfate solution and transfer into a clean 100 cm³ conical flask.
2. Use a 10 cm³ measuring cylinder to measure out 4 cm³ of 1.0 mol dm⁻³ hydrochloric acid solution.
3. Add the hydrochloric acid to the sodium thiosulfate solution in the conical flask and immediately start the stop clock.
4. Swirl the contents of the flask once to ensure mixing and place the flask onto the card marked with an X.
5. Observe the X from above and stop the stop clock as soon as the X disappears from view.
6. Measure the time taken (t) in seconds for the X to disappear from view. Record the time taken to the nearest whole number of seconds.
7. Rinse the conical flask thoroughly with deionised water before carrying out the next experiment.
8. Repeat the experiment for the other combinations shown in the table below, using a 10 cm³ measuring cylinder to measure the required volumes of deionised water.



Practical 1.1

Investigating the effect of changing concentration on the rate of a chemical reaction (spec ref 4.3.7)

Volume of $\text{Na}_2\text{S}_2\text{O}_3(\text{aq})$ / cm^3	Volume of water / cm^3	Concentration of $\text{Na}_2\text{S}_2\text{O}_3(\text{aq})$ / mol dm^{-3}	Volume of HCl / cm^3	Time taken / s	$\frac{1000}{t}$
10	0		4		
8	2		4		
6	4		4		
5	5		4		
4	6		4		
2	8		4		

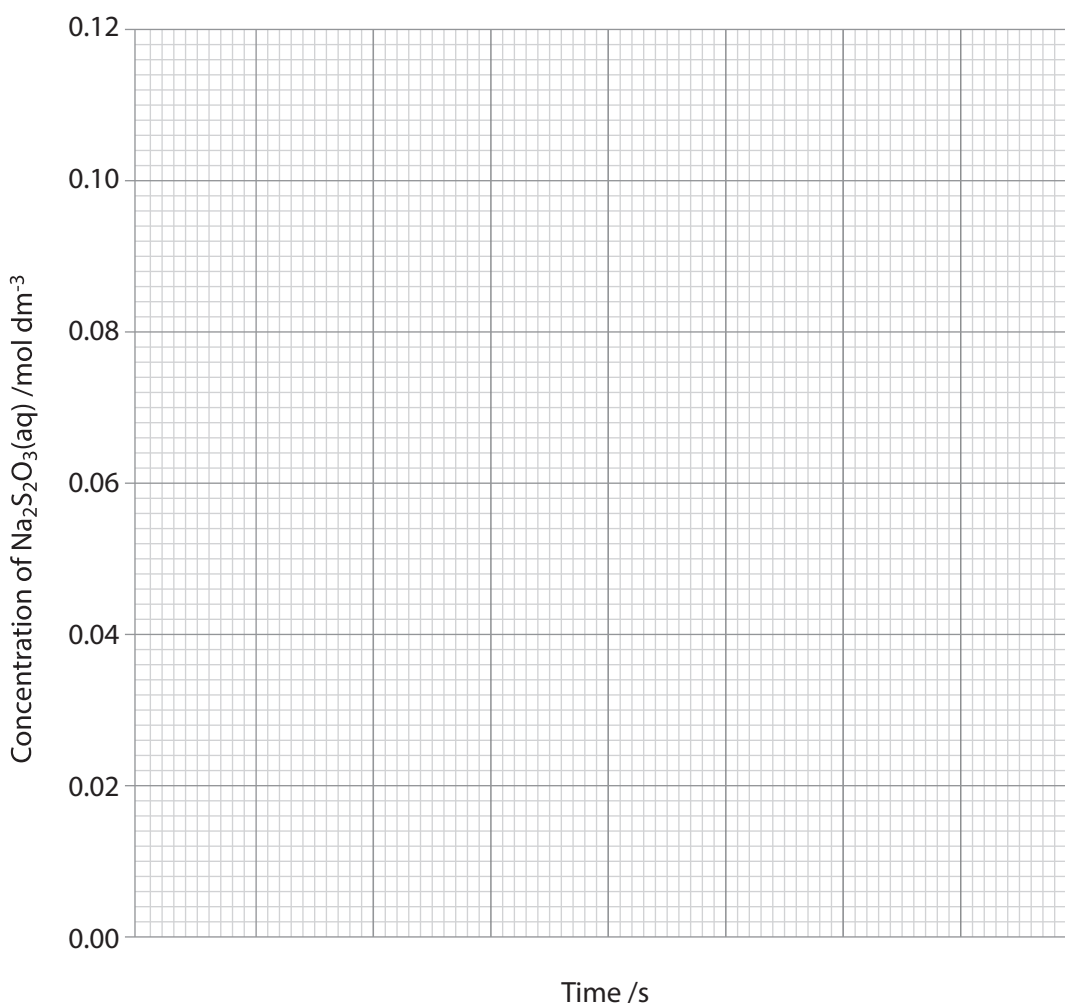
Results analysis

1. Calculate the number of moles of sodium thiosulfate added to each reaction mixture and use this to calculate the concentration of sodium thiosulfate solution in each reaction mixture. Put these values in the table.
2. Scale the horizontal axis appropriately for your results and plot a graph of concentration of sodium thiosulfate solution against time on the axes below.



Practical 1.1

Investigating the effect of changing concentration on the rate of a chemical reaction (spec ref 4.3.7)



As the reaction time is measured until the same, fixed amount of sulfur is produced, we can say that:

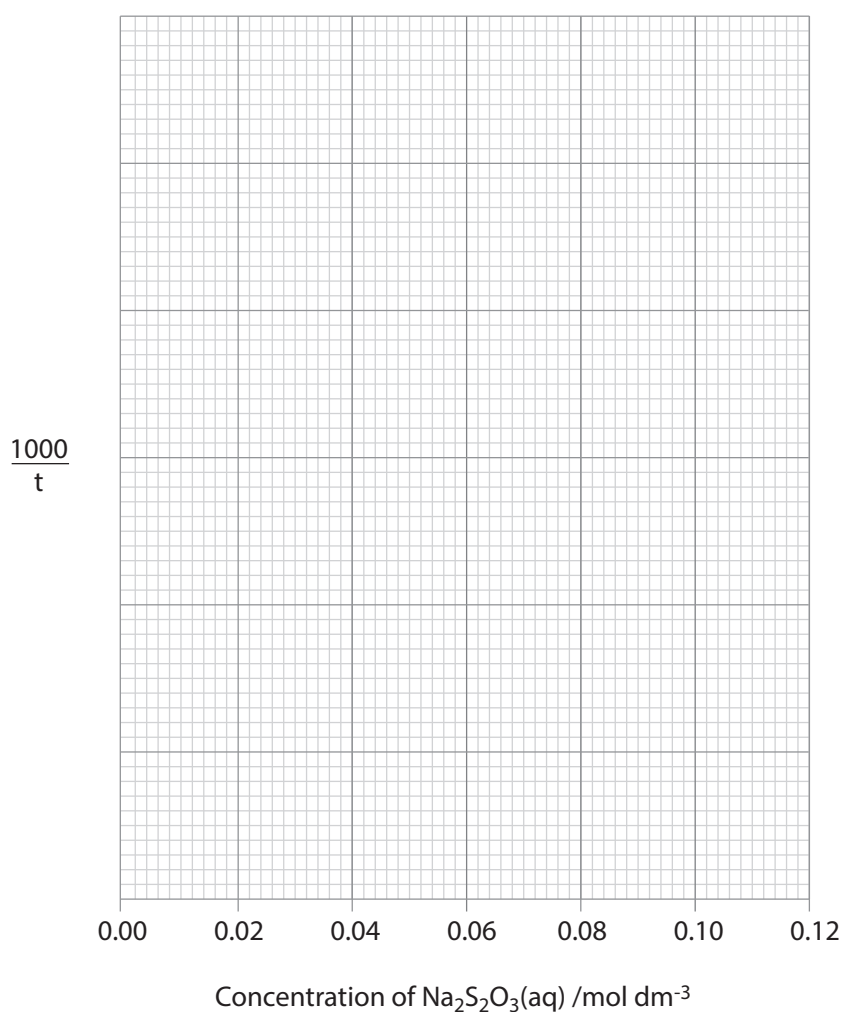
$$\text{Rate} = \frac{\text{amount of sulfur}}{\text{time}} \quad \text{or} \quad \text{rate} \propto \frac{1}{\text{time}}$$

3. Calculate and record the values of $\frac{1000}{t}$ for each reaction mixture.
4. Scale the vertical axis appropriately for your results and plot a graph of $\frac{1000}{t}$ against concentration of sodium thiosulfate solution on the axes below and draw a line of best fit through the points plotted.



Practical 1.1

Investigating the effect of changing concentration on the rate of a chemical reaction (spec ref 4.3.7)



Questions

1. What is the order of reaction with respect to sodium thiosulfate? Explain how your graph supports this.

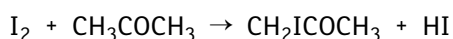


Practical 1.2

Investigating the effect of changing concentration on the rate of a chemical reaction (spec ref 4.3.7)

Introduction

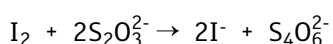
In this experiment the reaction between iodine and propanone is investigated; the reaction is catalysed by hydrogen ions from sulfuric acid:



The influence of the iodine on the reaction rate can be studied if the concentrations of propanone and hydrogen ions effectively remain constant during the reaction. This is achieved by using a large excess of both propanone and sulfuric acid in the starting reaction mixture. The results of the experiment will allow you to determine the order of reaction with respect to iodine.

$$\text{Rate} = k[\text{I}_2]^x$$

As the reaction proceeds, samples can be taken and quenched (this stops the reaction and allows the mixture to be analysed). The reaction mixture is then titrated with a standard solution of sodium thiosulfate to determine the amount of iodine remaining:



By determining how the concentration of iodine varies over a period of time, the order of reaction with respect to iodine can be determined.



Practical 1.2

Investigating the effect of changing concentration on the rate of a chemical reaction (spec ref 4.3.7)

Apparatus and materials

- propanone solution (1.0 mol dm^{-3})
- sulfuric acid (1.0 mol dm^{-3})
- iodine solution (0.02 mol dm^{-3})
- sodium thiosulfate solution (0.01 mol dm^{-3})
- starch solution
- sodium hydrogencarbonate
- $2 \times 25 \text{ cm}^3$ measuring cylinders
- 50 cm^3 measuring cylinder
- 250 cm^3 beaker
- $5 \times 250 \text{ cm}^3$ conical flasks
- 10.0 cm^3 graduated pipette
- pipette filler
- glass rod
- spatula
- stop clock
- safety glasses

Procedure

1. Measure 25 cm^3 of 1 mol dm^{-3} aqueous propanone using a measuring cylinder and add to a 250 cm^3 beaker.
2. Measure 25 cm^3 of 1 mol dm^{-3} sulfuric acid using a measuring cylinder and add to the 250 cm^3 beaker containing the aqueous propanone.
3. Measure 50 cm^3 of 0.02 mol dm^{-3} iodine solution using a measuring cylinder and add to the 250 cm^3 beaker containing the aqueous propanone and sulfuric acid. Start the stop clock and stir with a glass rod.
4. Using a pipette, withdraw a 10 cm^3 sample of the mixture and transfer it to a conical flask.
5. Stop the reaction by adding a spatula measure of sodium hydrogencarbonate and gently swirl the conical flask. Note the exact time at which the sodium hydrogencarbonate is added.
6. Titrate the remaining iodine present in the sample with 0.01 mol dm^{-3} sodium thiosulfate solution, using starch indicator.



Practical 1.2

Investigating the effect of changing concentration on the rate of a chemical reaction (spec ref 4.3.7)

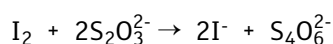
7. Continue to withdraw 10 cm³ samples at suitable time intervals (approximately every 5 minutes) and repeat steps 5 and 6. Note the exact time at which the sodium hydrogencarbonate is added to each sample.

Time sodium hydrogencarbonate added / s	Initial Burette Reading / cm ³	Final Burette Reading / cm ³	Titre / cm ³	Moles of sodium thiosulfate	Moles of iodine	Concentration of Iodine / mol dm ⁻³

Results analysis

The concentration of iodine in each quenched sample is determined as follows:

1. Calculate the number of moles of sodium thiosulfate required to react with the iodine in each sample;
2. Use the ratio in the equation to deduce the number of moles of iodine present in each sample;



3. Calculate the concentration of iodine in each 10 cm³ sample.
4. Plot a graph of concentration of iodine against time and use it to determine the order of reaction with respect to iodine.



Practical 1.2

Investigating the effect of changing concentration on the rate of a chemical reaction (spec ref 4.3.7)



Questions

1. What is the order of reaction with respect to iodine? Explain how your graph supports this.

2. What is the purpose of the sodium hydrogencarbonate? Explain how it acts, using an equation.



Practical 1.2

Investigating the effect of changing concentration on the rate of a chemical reaction (spec ref 4.3.7)

3. Explain the advantage of using starch as an indicator in this experiment.

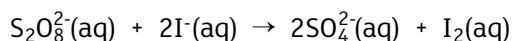


Practical 1.3

Investigating the effect of changing concentration on the rate of a chemical reaction (spec ref 4.3.7)

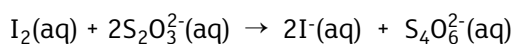
Introduction

Peroxodisulfate(VI) ions react with iodide ions to form iodine.



The iodine can be detected by its colour which can be enhanced by the addition of starch solution. The amounts of each of the reactants can be varied and the time it takes to produce a fixed amount of iodine can be measured.

One way of measuring the initial rate is to time how long it takes to produce a certain, small amount of iodine. This can be done by adding a fixed amount of sodium thiosulfate to the reaction mixture, which will react with a fixed amount of iodine.



When the sodium thiosulfate has been used up the blue-black iodine/starch colour will suddenly appear in solution. From the amount of thiosulfate added the amount of iodine produced can be calculated. This will allow the order of reaction with respect to iodide ions to be deduced.

Apparatus and materials

- boiling tubes
- plastic dropping pipettes
- potassium iodide solution (1.0 mol dm^{-3})
- potassium peroxodisulfate(VI) solution (0.04 mol dm^{-3})
- sodium thiosulfate solution (0.01 mol dm^{-3})
- deionised water
- starch solution
- stop clock
- safety goggles



Practical 1.3

Investigating the effect of changing concentration on the rate of a chemical reaction (spec ref 4.3.7)

Procedure

1. Measure out the quantities of potassium iodide solution, sodium thiosulfate solution, water and starch given for one of the mixtures in the table below using plastic dropping pipettes and add to a boiling tube.
2. Measure 2 cm³ of potassium peroxodisulfate(VI) solution using a plastic dropping pipette. Add to the boiling tube, gently shake and start the stop clock. Record the time taken for the blue-black colour to appear.

Mixture	Volume of potassium iodide solution / cm ³	Volume of water / cm ³	Volume of sodium thiosulfate solution / cm ³	Volume of starch solution cm ³	Volume of potassium peroxodisulfate(VI) solution / cm ⁻³	Time / s
1	5	0	2	1	2	
2	4	1	2	1	2	
3	3	2	2	1	2	
4	2	3	2	1	2	
5	1	4	2	1	2	

Results analysis

1. Calculate the number of moles of iodide ions added to each reaction mixture.
2. Calculate the concentration of iodide ions in each reaction mixture.
3. The amount of iodide ions in each reaction mixture is an excess; the amount of iodine produced is therefore determined by the amount of peroxodisulfate(VI) in each reaction mixture. Calculate the number of moles of peroxodisulfate(VI) added to each reaction mixture and therefore the number of moles of iodine produced in each reaction.
4. The added thiosulfate ions react with a fixed amount of iodine, delaying the appearance of the blue-black colour. Calculate the number of moles of thiosulfate added to each reaction mixture and the number of moles of iodine that react with the thiosulfate.
5. The initial rate of reaction can be measured as the concentration of iodine produced per second (mol dm⁻³ s⁻¹). For each reaction mixture, divide the number of moles of iodine that react with thiosulfate by the time taken for the appearance of the blue-black colour.
6. Plot a graph of rate against concentration of iodide ions (calculated in 2).



Practical 1.3

Investigating the effect of changing concentration on the rate of a chemical reaction (spec ref 4.3.7)

Mixture	Moles of iodide added	Concentration of iodide / mol dm ⁻³	Moles of peroxodisulfate(VI) added	Moles of iodine produced	Moles of thiosulfate added	Moles of iodine reacting with thiosulfate	Initial rate / mol dm ⁻³ s ⁻¹
1							
2							
3							
4							
5							





Practical 1.3

Investigating the effect of changing concentration on the rate of a chemical reaction (spec ref 4.3.7)

Questions

1. What is the order of reaction with respect to iodide? Explain how your graph supports this.

2. The method used is known as the 'iodine clock method' and is used to determine the initial rate of reaction. Using the maximum amount of iodine produced in each reaction mixture and amount that reacts with thiosulfate, calculate the extent of the reaction that is studied. Express your answer as a percentage.

3. Calculate the value for the rate constant for the reaction using the gradient of your graph. Include the units in your answer.



Practical 2.1

Making buffer solutions and investigating their pH values (spec ref 4.5.5)

Introduction

A buffer solution is one which resists changes in pH on addition of small amounts of acid or alkali. There are two types:

Acidic Buffer (pH < 7)

These are made from a weak acid and salt of its conjugate base, for example ethanoic acid and sodium ethanoate.

- If H^+ ions are added, they are removed by CH_3COO^- :
$$\text{CH}_3\text{COO}^- + \text{H}^+ \rightarrow \text{CH}_3\text{COOH}$$
- If OH^- ions are added, they are removed by CH_3COOH :
$$\text{CH}_3\text{COOH} + \text{OH}^- \rightarrow \text{CH}_3\text{COO}^- + \text{H}_2\text{O}$$

Alkaline Buffer (pH > 7)

These are made from a weak base and salt of its conjugate acid, for example ammonia and ammonium chloride.

- If H^+ ions are added, they are removed by NH_3 :
$$\text{NH}_3 + \text{H}^+ \rightarrow \text{NH}_4^+$$
- If OH^- ions are added, they are removed by NH_4^+ :
$$\text{NH}_4^+ + \text{OH}^- \rightarrow \text{NH}_3 + \text{H}_2\text{O}$$

The quantities of the two components of a buffer solution must be large relative to the amount of acid or alkali added in order for the pH to maintain relatively unchanged. Buffer solutions are used in a range of cosmetic products and biological systems.



Practical 2.1

Making buffer solutions and investigating their pH values (spec ref 4.5.5)

Apparatus and materials

- 5 × 100 cm³ beakers
- 2 × 25 cm³ measuring cylinders
- 10 cm³ measuring cylinder
- ethanoic acid (0.5 mol dm⁻³)
- potassium ethanoate solution (0.5 mol dm⁻³)
- methanoic acid (0.5 mol dm⁻³)
- potassium methanoate solution (0.5 mol dm⁻³)
- ammonia solution (0.5 mol dm⁻³)
- ammonium chloride (0.5 mol dm⁻³)
- hydrochloric acid (0.5 mol dm⁻³)
- sodium hydroxide solution (0.5 mol dm⁻³)
- pH meter/Universal indicator paper (1-14)
- glass rod
- deionised water

Procedure

1. Make up three buffer solutions by mixing 25 cm³ portions of the 0.5 mol dm⁻³ solutions in the pairs listed below. Measure out each solution using a measuring cylinder and transfer each pair of solutions into a 100 cm³ beaker. Stir with a glass rod.
Buffer 1: ethanoic acid and potassium ethanoate solution
Buffer 2: methanoic acid and potassium methanoate solution
Buffer 3: ammonia solution and ammonium chloride solution
2. Measure the pH of each buffer solution using a pH meter and Universal indicator paper.
3. Transfer two separate 10 cm³ portions of buffer 1 to two 100 cm³ beakers. Add 1 cm³ of hydrochloric acid to one of the beakers and 1 cm³ of sodium hydroxide solution to the other beaker. Stir with a glass rod and record the pH of each solution with a pH meter and Universal indicator paper.
4. Repeat step 3 for the other two buffer solutions.



Practical 2.1

Making buffer solutions and investigating their pH values (spec ref 4.5.5)

Buffer	pH	pH on addition of HCl(aq)	pH on addition of NaOH(aq)
1			
2			
3			

Questions

1. Explain, with relevant equations, why the pH does not significantly change when hydrochloric acid or sodium hydroxide solution is added to each of the buffer solutions.



Practical 2.1

Making buffer solutions and investigating their pH values (spec ref 4.5.5)

2. Calculate the pH (to two decimal places) of a buffer solution made by adding 1.42 g of potassium methanoate to 50 cm³ of 0.111 mol dm⁻³ methanoic acid. (K_a methanoic acid = 1.78×10^{-4} mol dm⁻³).

3. Calculate the mass of sodium ethanoate that must be added to 100 cm³ of 1.0 mol dm⁻³ ethanoic acid to make a buffer solution of pH 4.38. (K_a ethanoic acid = 1.8×10^{-5} mol dm⁻³).



Practical 2.2

Determining the shape of a titration curve (spec ref: 4.5.6)

Introduction

In a neutralisation reaction, an acid and base react to form a salt and water. The change in pH as the acid is added to the base (or vice versa) can be tracked using a pH meter or Universal indicator solution and plotted to give a titration curve.

Apparatus and materials

- hydrochloric acid (0.1 mol dm^{-3})
- sodium hydroxide solution (0.1 mol dm^{-3})
- pH meter/Universal indicator solution
- burette
- pipette
- pipette filler
- 250 cm^3 conical flask
- deionised water

Procedure

1. Using a pipette and pipette filler, transfer 25.0 cm^3 of 0.1 mol dm^{-3} hydrochloric acid into a conical flask. Record the pH using the pH meter (or alternatively, add a few drops of Universal indicator solution and swirl. Record the pH).
2. Fill a burette with 0.1 mol dm^{-3} sodium hydroxide solution. Add sodium hydroxide solution, 2 cm^3 at a time, to the conical flask. Swirl the flask and record the pH after each addition.
3. Between 24.0 cm^3 and 26.0 cm^3 , add sodium hydroxide solution 0.5 cm^3 at a time.
4. Continue to add sodium hydroxide solution, 2 cm^3 at a time, between 26.0 cm^3 and 40.0 cm^3 . Swirl the flask and record the pH after each addition.



Practical 2.2

Determining the shape of a titration curve
(spec ref: 4.5.6)

Volume of sodium hydroxide / cm ³	pH
0.0	
2.0	
4.0	
6.0	
8.0	
10.0	
12.0	
14.0	
16.0	
18.0	
20.0	
22.0	
24.0	
24.5	
25.0	
25.5	
26.0	
28.0	
30.0	
32.0	
34.0	
36.0	
38.0	
40.0	

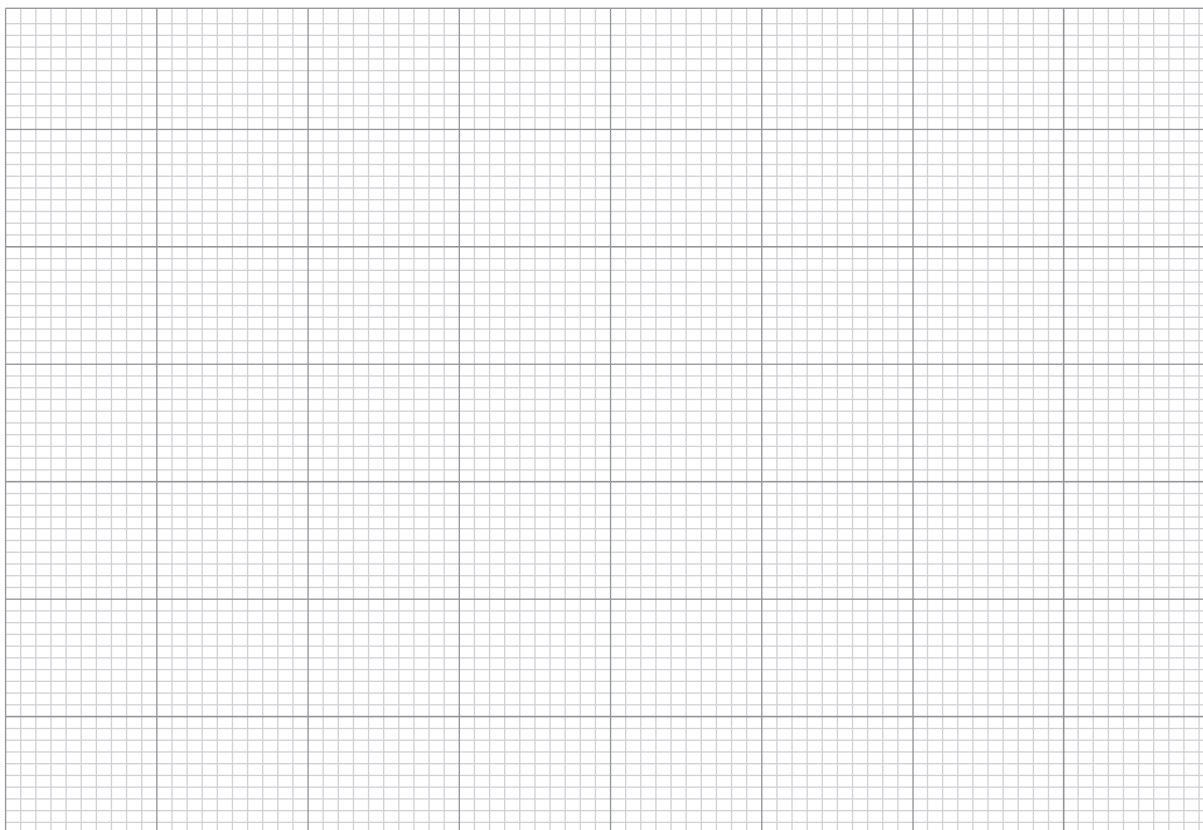


Practical 2.2

Determining the shape of a titration curve (spec ref: 4.5.6)

Results analysis

1. Plot a graph of pH against volume of sodium hydroxide solution. Join the points with a smooth curve.





Practical 2.2

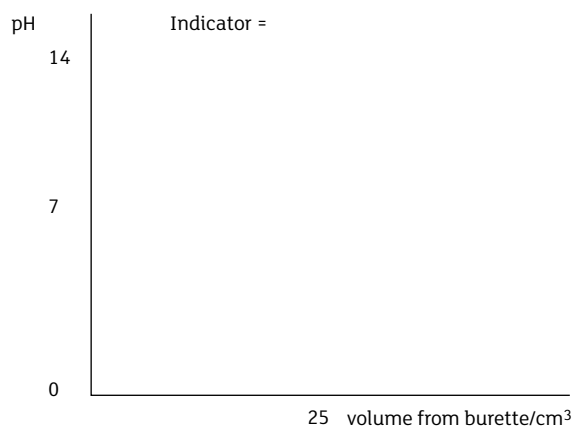
Determining the shape of a titration curve (spec ref: 4.5.6)

Questions

1. Sketch each of the following pH curves on the grids shown and name a suitable indicator.

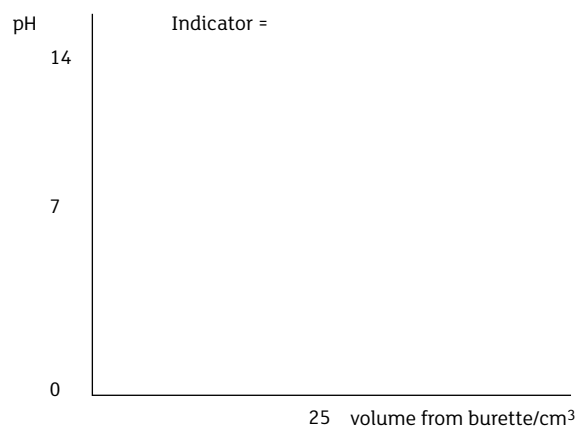
(1) Flask 25 cm^3 $0.10 \text{ mol dm}^{-3} \text{ H}_2\text{SO}_4$

Burette 50 cm^3 $0.20 \text{ mol dm}^{-3} \text{ NaOH}$



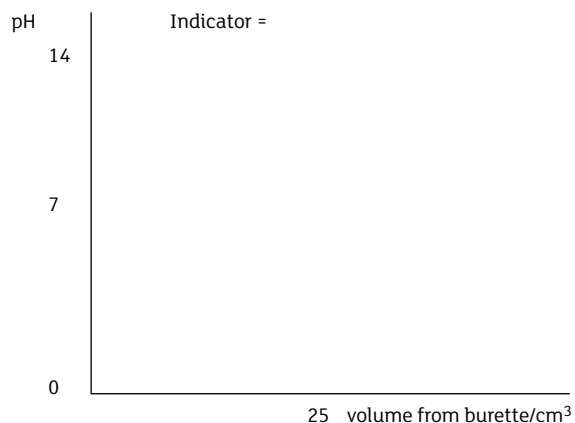
(2) Flask 20 cm^3 $1.00 \text{ mol dm}^{-3} \text{ NH}_3$

Burette 50 cm^3 $0.50 \text{ mol dm}^{-3} \text{ HCl}$



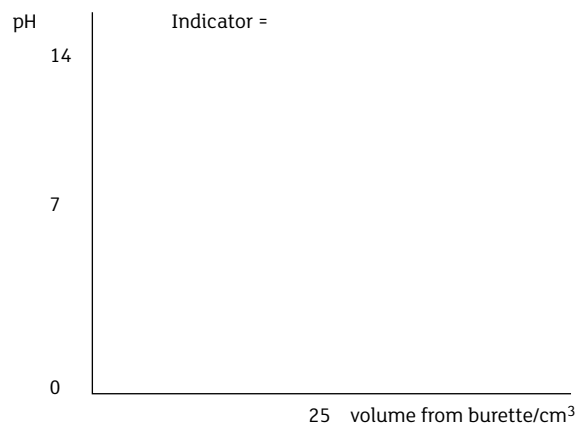
(3) Flask 20 cm^3 $0.20 \text{ mol dm}^{-3} \text{ CH}_3\text{COOH}$

Burette 50 cm^3 $0.20 \text{ mol dm}^{-3} \text{ NaOH}$



(4) Flask 25 cm^3 $0.50 \text{ mol dm}^{-3} \text{ NH}_3$

Burette 50 cm^3 1.00 mol dm^{-3} methanoic acid





Practical 2.3

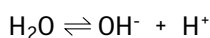
Determining the pH of salts (spec ref: 4.5.8)

Introduction

The pH at the end point of an acid/base titration is not always 7 as it depends on the relative strengths of the acid and base used. The salt produced in an acid-base reaction will therefore not necessarily be neutral in solution.

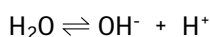
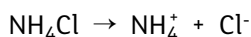
1. Salt of a strong acid and a strong base eg. NaCl

This type of salt dissolves to form a neutral solution. The ions present are those of a strong acid and strong base; Na^+ and Cl^- ions do not react with water and so the solution is neutral.



2. Salt of a strong acid and a weak base eg. NH_4Cl

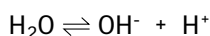
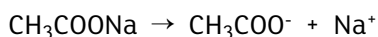
This type of salt forms an acidic solution when added to water.



The ammonium ions react reversibly with hydroxide ions as they are ions of a weak base. $[\text{H}^+] > [\text{OH}^-]$ so the salt solution is acidic.

3. Salt of a weak acid and a strong base eg. CH_3COONa

This type of salt forms an alkaline solution when added to water.



The ethanoate ions react reversibly with hydrogen ions as they are ions of a weak acid. $[\text{H}^+] < [\text{OH}^-]$ so salt solution is alkaline.

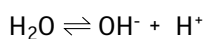
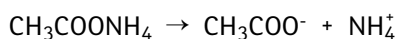


Practical 2.3

Determining the pH of salts (spec ref: 4.5.8)

4. Salt of a weak acid and a weak base eg. $\text{CH}_3\text{COONH}_4$

The salt solution formed will be approximately neutral.



The ethanoate ions react reversibly with hydroxide ions and the ammonium ions react reversibly with hydrogen ions. This results in a solution which is approximately neutral, although it depends on how weak the acid and base are.

Apparatus and materials

- sodium chloride
- sodium ethanoate
- ammonium chloride
- ammonium ethanoate
- pH meter
- 250 cm³ deionised water
- 50 cm³ measuring cylinder
- 100 cm³ beakers
- weighing boats
- glass rod
- access to a mass balance (2 d.p)



Practical 2.3

Determining the pH of salts (spec ref: 4.5.8)

Procedure

1. Calculate the mass of each salt required to make 50 cm³ of 0.5 mol dm⁻³ solutions of each salt.
2. Measure four separate 50 cm³ portions of deionised water using a measuring cylinder and transfer each portion to a separate beaker.
3. Weigh the calculated mass of each salt and transfer to each beaker. Stir to dissolve.
4. Record the pH of the salt solution with the pH meter. Rinse the pH probe with deionised water between readings.

Salt	pH
Sodium chloride	
Sodium ethanoate	
Ammonium chloride	
Ammonium ethanoate	

Questions

1. Explain the result obtained for each salt, using relevant equations.



Practical 2.3

Determining the pH of salts (spec ref: 4.5.8)

2. When each of the following salts is dissolved in water, is the pH greater than, less than, or equal to 7? Explain your answers.

- potassium cyanide, KCN
- sodium methanoate, HCOONa
- methylammonium chloride, CH₃NH₃Cl
- ammonium sulfate, (NH₄)₂SO₄
- potassium sulfate, K₂SO₄
- sodium carbonate, Na₂CO₃
- sodium fluoride, NaF

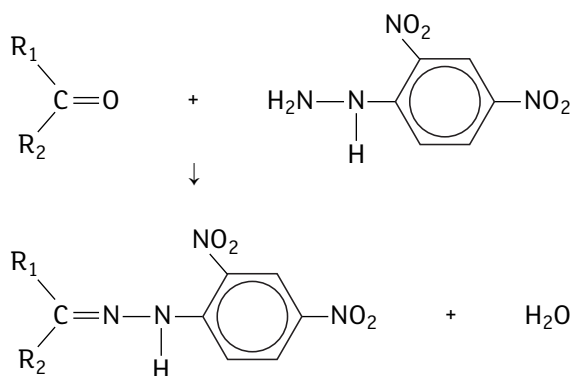


Practical 3.1

Preparation of 2,4-dinitrophenylhydrazones (spec ref: 4.7.8)

Introduction

Carbonyl compounds react with 2,4-dinitrophenylhydrazine in a condensation reaction to form yellow/orange solid 2,4-dinitrophenylhydrazone derivatives. The melting point of the derivatives can be used to identify the carbonyl compound used.



You are provided with an unknown carbonyl compound X. You will prepare a derivative of the compound with 2,4-dinitrophenylhydrazine, recrystallise it and determine its melting point in order to identify the aldehyde or ketone X.

Apparatus and materials

- 100 cm³ beaker
- 250 cm³ beaker
- glass rod
- plastic dropping pipettes
- 2,4-dinitrophenylhydrazine solution
- aldehyde or ketone labelled X
- Büchner funnel and flask
- spatula
- ethanol
- filter paper
- access to a drying oven
- a capillary tube sealed at one end
- access to melting point apparatus

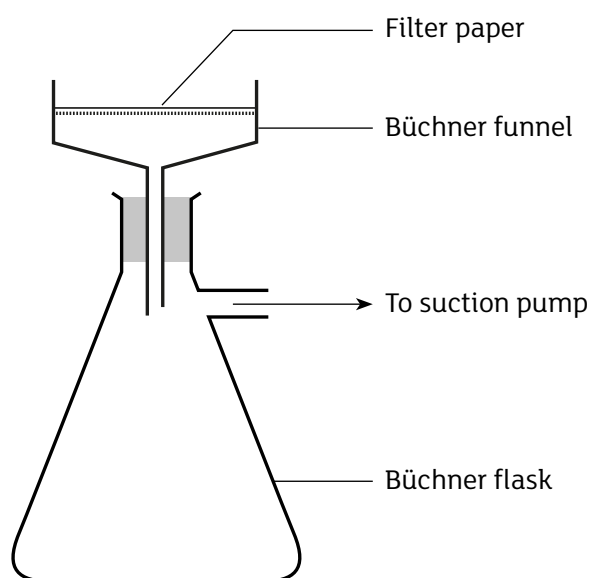


Practical 3.1

Preparation of 2,4-dinitrophenylhydrazones (spec ref: 4.7.8)

Procedure

1. Use a plastic dropping pipette to add 5 cm³ of 2,4-dinitrophenylhydrazine solution to a 100 cm³ beaker.
2. Use a plastic dropping pipette to add 10 drops of the unknown carbonyl compound X to the 100 cm³ beaker and stir with the glass rod.
3. Filter the precipitate using suction filtration.



4. Transfer the crude product to a 100 cm³ beaker placed in a 250 cm³ beaker containing hot water.
5. Dissolve the crude product in the minimum volume of hot ethanol. Filter while hot using gravity filtration.
6. When the solid has disappeared, cool the solution in a 250 cm³ beaker containing iced water.
7. Filter the crystals using suction filtration and wash with a small portion of cold ethanol.
8. Transfer the crystals to a sheet of filter paper and place in a drying oven overnight.
9. Transfer a few crystals to a capillary tube sealed at one end and use the melting point apparatus to determine their melting point.



Practical 3.1

Preparation of 2,4-dinitrophenylhydrazones (spec ref: 4.7.8)

Questions

1. Over what temperature range did the crystals melt? How does this indicate the purity of the sample?

2. Use the melting point of the hydrazine derivative to identify the carbonyl compound X. Use the table below as a guide.

Aldehyde/Ketone	Melting point of hydrazone (°C)
Ethanal	164
Propanal	156
Propanone	128
Butanone	115

3. Write an equation for the reaction between the identified carbonyl compound and 2,4-dinitrophenylhydrazine.

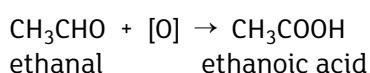


Practical 3.2

Using Fehling's solution and Tollens' reagent to distinguish between aldehydes and ketones (spec ref 4.7.9)

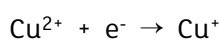
Introduction

Aldehydes are oxidised to carboxylic acids using either Fehling's solution or Tollens' reagent. For example, ethanal is readily oxidised to ethanoic acid:

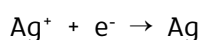


Ketones are not oxidised by either oxidising agent and so these reactions can be used to distinguish between and aldehyde and ketone.

Fehling's solution is a mixture of copper(II) sulfate and sodium potassium tartrate in sodium hydroxide solution. This blue solution will oxidise aldehydes and will itself be reduced to give a red precipitate of copper(I) oxide, Cu_2O . This can be represented by the following half equation:



Tollens' reagent is usually prepared when required and is an ammoniacal solution of silver(I). It contains the diamminesilver(I) ion, $[\text{Ag}(\text{NH}_3)_2]^+$. The colourless solution produces a silver mirror when warmed with an aldehyde. This can be represented by the following half equation:



Apparatus and materials

- test tubes
- 250 cm³ beaker
- plastic dropping pipettes
- Fehling's solution 1
- Fehling's solution 2
- silver nitrate solution
- ammonia solution
- ethanal
- propanone



Practical 3.2

Using Fehling's solution and Tollens' reagent to distinguish between aldehydes and ketones (spec ref 4.7.9)

Procedure

(a) Using Fehling's solution

1. Use a plastic dropping pipette to add 1 cm^3 of Fehling's solution 1 to a test tube and then add 1 cm^3 of Fehling's solution 2.
2. Use a plastic dropping pipette to add between 5–10 drops of ethanal to the test tube. Shake the tube gently and place in a beaker of warm water for 5–10 minutes. Note your observations.
3. Repeat using propanone.

(B) Using Tollens' reagent

1. Use a plastic dropping pipette to add 1 cm^3 of silver nitrate solution to a test tube.
2. Use a plastic dropping pipette to add ammonia solution dropwise until the cloudy precipitate just dissolves.
3. Use a plastic dropping pipette to add 2 drops of ethanal to the test tube. Shake the tube gently and place in a beaker of boiling water for 5 minutes. Note your observations.
4. Repeat using propanone.

Carbonyl compound	Observation with Fehling's solution	Observation with Tollens' reagent
Ethanal		
Propanone		



Practical 3.2

Using Fehling's solution and Tollens' reagent to distinguish between aldehydes and ketones (spec ref 4.7.9)

Questions

1. Acidified potassium dichromate(VI) can also be used to oxidise aldehydes. State the colour change observed.

2. Write a half-equation for the reduction of dichromate(VI) to chromium(III).

3. Explain why the observations obtained with 2,4-dinitrophenylhydrazine cannot be used to distinguish between an aldehyde and a ketone.

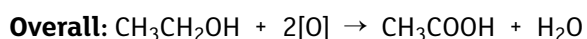
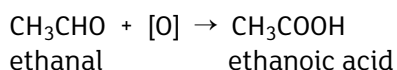
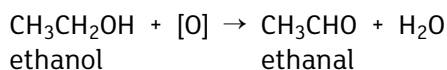


Practical 4.1

Preparing a carboxylic acid from an alcohol (spec ref 4.8.3)

Introduction

Alcohols are oxidised to aldehydes which can be further oxidised to carboxylic acids. Heating an alcohol under reflux with acidified dichromate(VI) solution produces the corresponding carboxylic acid. For example, using ethanol:



Apparatus and materials

- safety goggles
- gloves
- 10 cm³ measuring cylinder
- spatula
- plastic dropping pipette
- funnel
- pear-shaped flask, still head, condenser with rubber tubing attached, receiver adapter with vent
- 100 cm³ conical flask
- retort stands, bosses and clamps
- Bunsen burner, tripod, gauze, heatproof mat
- weighing boat
- anti-bumping granules
- thermometer (0–200 °C) and adapter
- sulfuric acid (1 mol dm⁻³)
- concentrated sulfuric acid
- 5.0 g sodium dichromate(VI)
- ethanol
- access to a mass balance (1 d.p.)
- Universal indicator paper
- sodium carbonate

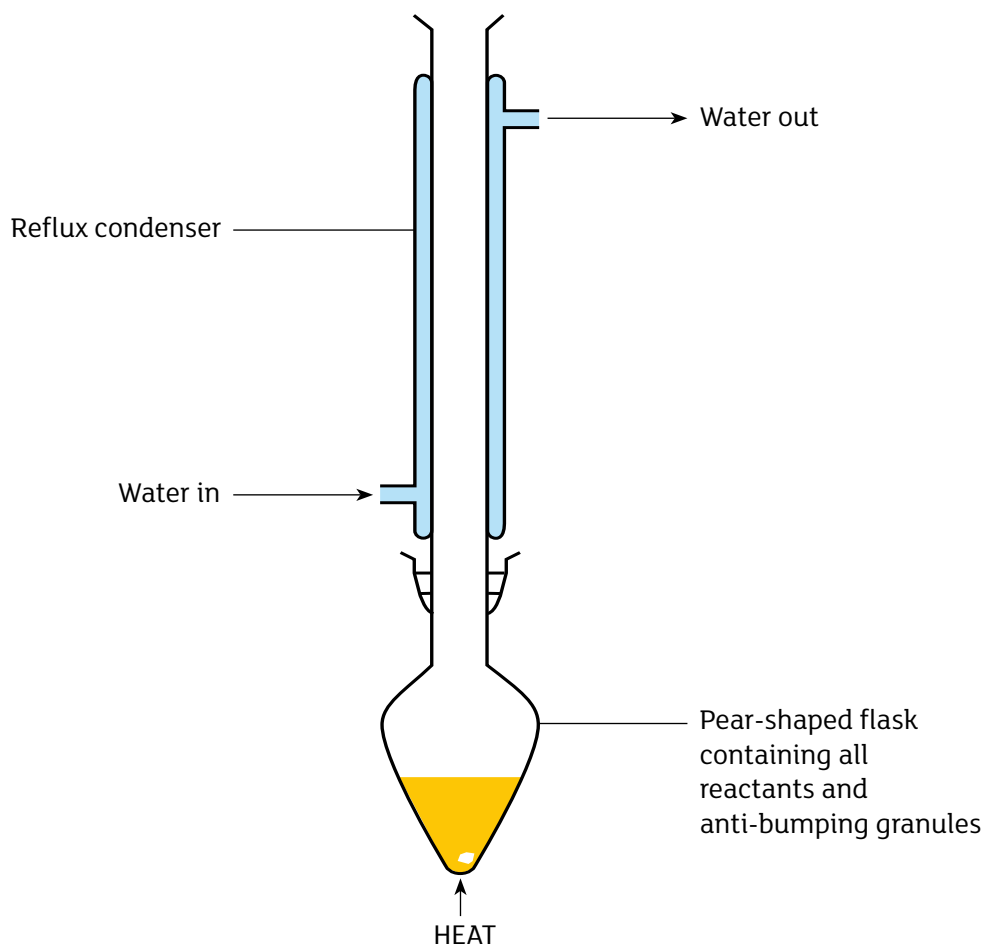


Practical 4.1

Preparing a carboxylic acid from an alcohol (spec ref 4.8.3)

Procedure

1. Using a measuring cylinder add 10 cm^3 of 1 mol dm^{-3} sulfuric acid into a 50 cm^3 pear-shaped flask.
2. Weigh 5.0 g of sodium dichromate(VI) and add to the flask followed by 1 spatula of anti-bumping granules. Swirl the flask until all the oxidising agent has dissolved.
3. Using a measuring cylinder, slowly add 2 cm^3 of concentrated sulfuric acid with care and then attach a condenser to the flask. If necessary, cool the flask under a running tap of cold water as the concentrated sulfuric acid is added.
4. Using a plastic dropping pipette, add 1 cm^3 ethanol, a few drops at a time, down the reflux condenser. Allow for the reaction to subside after each addition before adding more.
5. Set up a heat mat, tripod, wire gauze and Bunsen burner beneath the pear-shaped flask. Leave a small gap between the wire gauze and the bottom of the pear-shaped flask.
6. Gently reflux the contents of the pear shaped flask for 15–20 minutes.

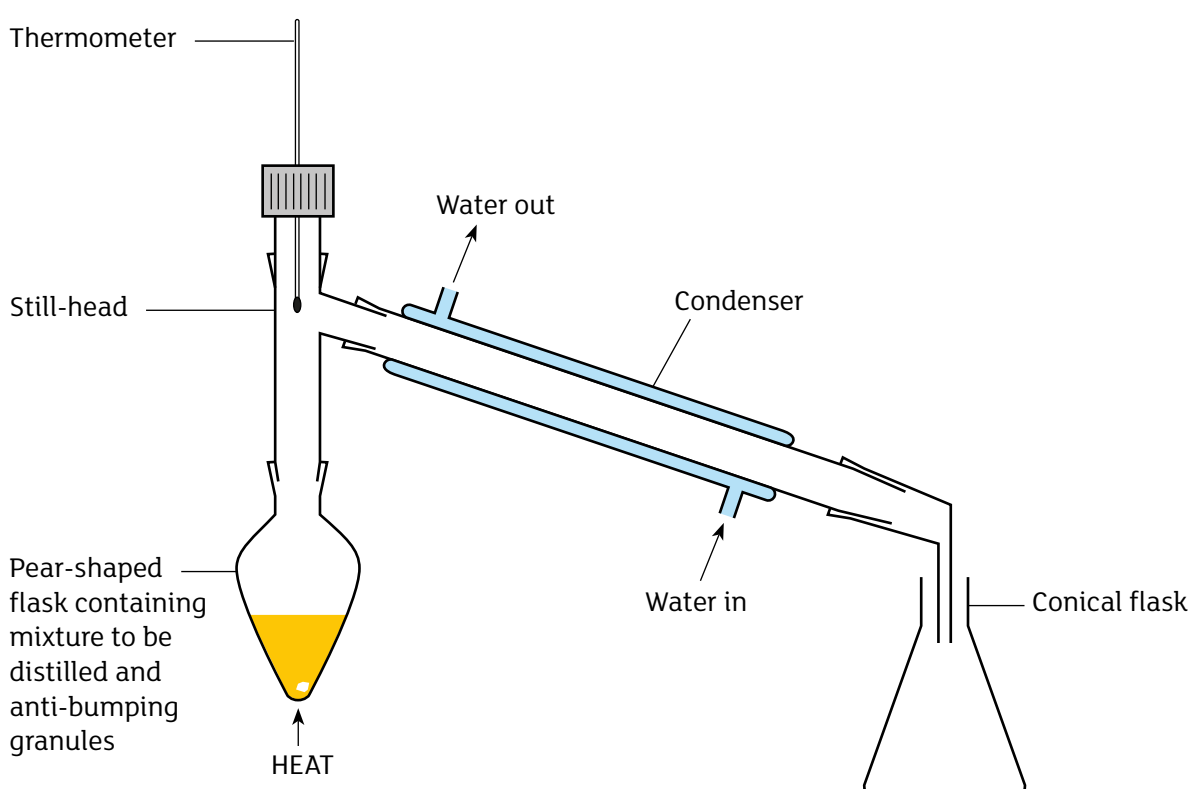




Practical 4.1

Preparing a carboxylic acid from an alcohol (spec ref 4.8.3)

7. Allow the flask to cool then rearrange the apparatus for simple distillation.



8. Distil off about 3–4 cm³ of liquid and perform the following tests:

- Smell cautiously
- Test with Universal indicator paper
- Add a spatula measure of solid sodium carbonate

Test	Observations
Smell	
Universal indicator paper	
Sodium carbonate	



Practical 4.1

Preparing a carboxylic acid from an alcohol (spec ref 4.8.3)

Questions

1. Explain why the concentrated sulfuric acid should be added slowly and with care.

2. Explain the purpose of the anti-bumping granules.

3. Explain how the results of the tests completed on the distillate confirm it contains ethanoic acid.

4. Describe what changes you would make to the method in order to obtain ethanal rather than ethanoic acid.



Practical 4.2

Carrying out test tube reactions of a carboxylic acid with sodium carbonate, sodium hydroxide and aqueous ammonia and measuring pH changes (spec ref 4.8.5)

Introduction

Carboxylic acids react as dilute weak acids with sodium carbonate, sodium hydroxide and ammonia. The salts produced are known as carboxylate salts.

Apparatus and materials

- safety goggles
- plastic dropping pipettes
- test tubes
- test tube rack
- spatula
- ethanoic acid (1 mol dm^{-3})
- sodium carbonate
- sodium hydroxide solution (1 mol dm^{-3})
- ammonia solution (1 mol dm^{-3})
- Universal indicator paper (1–14)
- thermometer (0–100 °C)
- delivery tube with bung
- limewater

Procedure

1. Using a plastic dropping pipette add 1 cm^3 of ethanoic acid to a test tube.
2. Test with Universal indicator paper and record the pH.
3. Add a spatula measure of sodium carbonate to the test tube and record any observations. Bubble any gas evolved through limewater.
4. When the reaction subsides record the pH using Universal indicator paper.
5. Using a plastic dropping pipette add 1 cm^3 of ethanoic acid to a test tube.
6. Test with Universal indicator paper and record the pH.
7. Record the temperature of the acid with a thermometer.
8. Add 1 cm^3 of sodium hydroxide solution to the test tube and note any observations. Record the maximum temperature reached.
9. When the reaction subsides test with Universal indicator paper and record the pH.
10. Repeat the experiment using ammonia solution instead of sodium hydroxide solution.



Practical 4.2

Carrying out test tube reactions of a carboxylic acid with sodium carbonate, sodium hydroxide and aqueous ammonia and measuring pH changes (spec ref 4.8.5)

	Observations
Reaction with sodium carbonate	
Reaction with sodium hydroxide	
Reaction with ammonia	

Questions

1. Write equations for reaction of ethanoic acid with sodium carbonate, sodium hydroxide and ammonia.

2. Write an equation for the reaction which occurs when carbon dioxide is bubbled through limewater.

3. Describe another chemical test which could be used to identify the presence of an -OH group in a carboxylic acid.

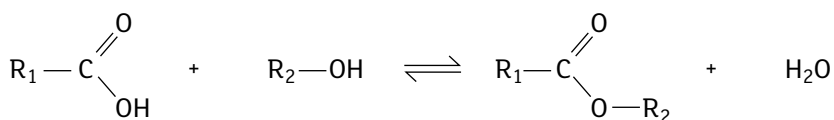


Practical 5.1

Preparing a liquid ester from a carboxylic acid and an alcohol (spec ref 4.9.5)

Introduction

Esters can be made by heating, under reflux, a mixture of an alcohol, a carboxylic acid and a few drops of concentrated sulphuric acid. This reaction is reversible and is known as a condensation reaction as water is also produced:



The concentrated sulfuric acid acts as a catalyst; it also removes water as it is formed which drives the equilibrium to the right-hand side. This increases the yield of ester produced.

In this practical you will prepare and purify the ester ethyl ethanoate.

Apparatus and materials

- safety glasses & protective gloves
- 50 cm³ pear-shaped flask, still head, condenser with rubber tubing attached, receiver adapter, 100 cm³ conical flask to collect distillate
- tripod, heat mat, wire gauze, Bunsen burner
- 10 cm³ measuring cylinder
- 25 cm³ measuring cylinder
- spatula
- plastic dropping pipette
- 2 retort stands, bosses & clamps
- 250 cm³ beaker
- separating funnel & stopper
- 100 cm³ conical flasks
- thermometer (0–100 °C) and adapter
- anti-bumping granules
- ethanol
- glacial ethanoic acid
- concentrated sulfuric acid
- sodium carbonate solution
- anhydrous sodium sulfate

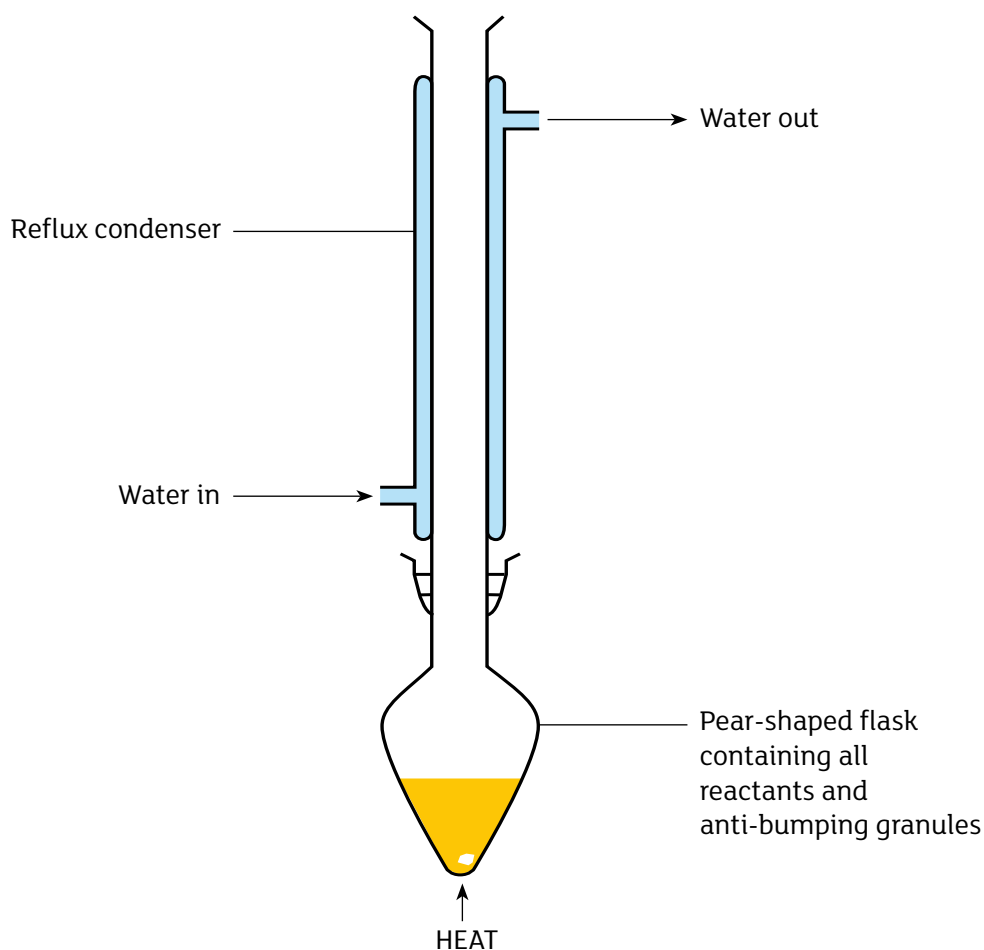


Practical 5.1

Preparing a liquid ester from a carboxylic acid and an alcohol (spec ref 4.9.5)

Procedure

1. Add a spatula measure of anti-bumping granules to a 50 cm³ pear-shaped flask.
2. In a fume-cupboard, add 10 cm³ ethanol, 12 cm³ glacial ethanoic acid and 15 drops of concentrated sulfuric acid to the pear-shaped flask.
3. Place a 250 cm³ beaker containing some water on a tripod and gauze over a Bunsen burner.
4. Clamp the pear-shaped flask in the beaker of water so that the reaction mixture is below the water line.
5. Connect the condenser to the pear-shaped flask and clamp the condenser.
6. Gently boil the reaction mixture in the water bath for 15–20 minutes. Turn off the Bunsen burner and cool the mixture by removing the hot water bath.

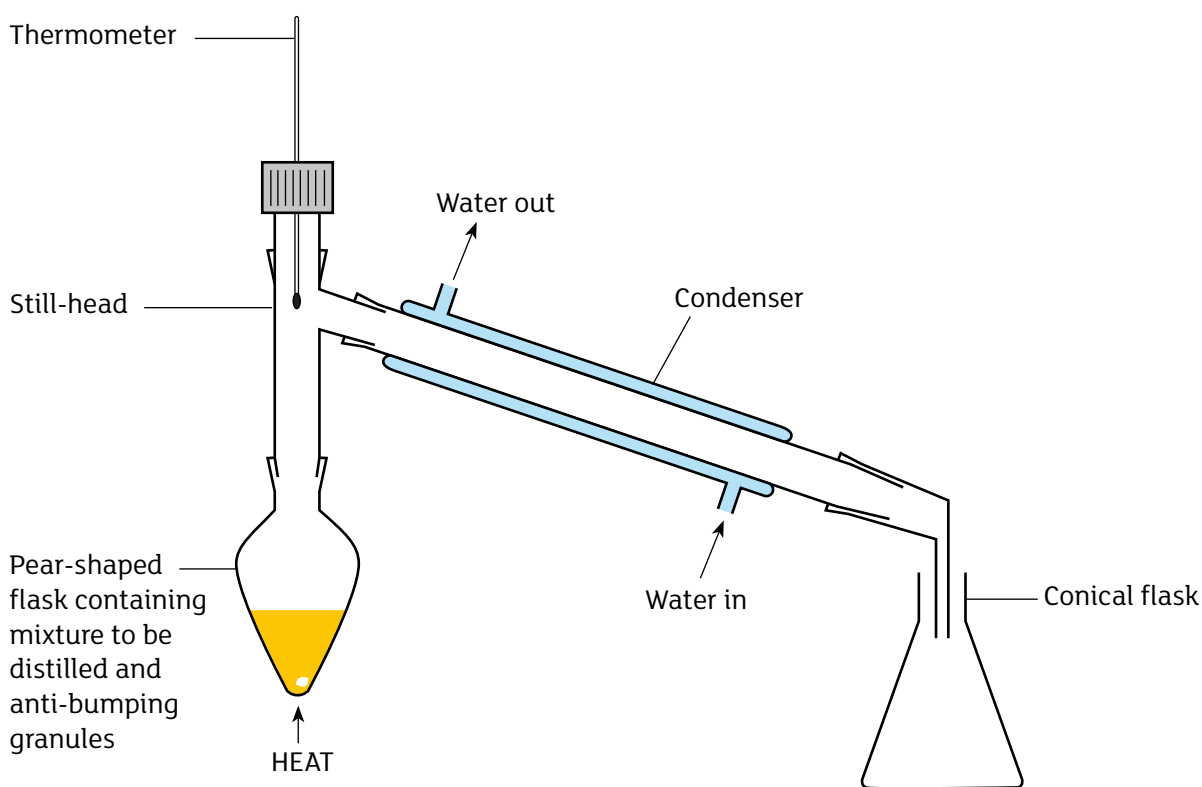




Practical 5.1

Preparing a liquid ester from a carboxylic acid and an alcohol (spec ref 4.9.5)

7. Rearrange the apparatus for distillation, heat the mixture gently and collect the distillate around 2 °C either side of the boiling point of the ester (77–78 °C).



8. Transfer the distillate (impure ester) into a separating funnel.
9. Add 10 cm³ of sodium carbonate solution to the impure ester in the separating funnel, stopper and shake gently. Release the pressure by opening the tap.
10. Remove the stopper, allow the layers to separate and discard the lower, aqueous layer (the ester is less dense than water).
11. Repeat steps 8 and 9 if needed until no more effervescence is observed.
12. Transfer the ester to a 100 cm³ conical flask.
13. Add a spatula measure of anhydrous sodium sulfate to the ester in the conical flask and shake. Repeat until the ester changes from cloudy to clear.
14. Decant the ethyl ethanoate into a clean, dry 50 cm³ pear-shaped flask.
15. Add a spatula measure of anti-bumping granules to the ester in the pear-shaped flask.



Practical 5.1

Preparing a liquid ester from a carboxylic acid and an alcohol (spec ref 4.9.5)

16. Place a 250 cm³ beaker containing some water on a tripod and gauze over a Bunsen burner.
17. Clamp the pear shaped flask in the beaker of water.
18. Arrange the apparatus for distillation and gently heat, collecting the ester at its boiling point (77–78 °C). Note that a fraction may distill off at 35–40 °C. This is an organic impurity (ether) which should be collected and discarded.

Questions

1. Write an equation for the reaction between ethanol and ethanoic acid.

2. State two functions of concentrated sulfuric acid in this experiment.

3. Explain, using two equations, the function of sodium carbonate solution in this experiment.

4. Explain how you could determine which layer is the aqueous layer if the density of the ester is unknown.

5. Ethoxyethane (ether), (C₂H₅)₂O, can be formed in a side reaction during the preparation of ethyl ethanoate. Ethanol can undergo dehydration in the presence of concentrated sulfuric acid to form ethoxyethane. Write an equation for this reaction.

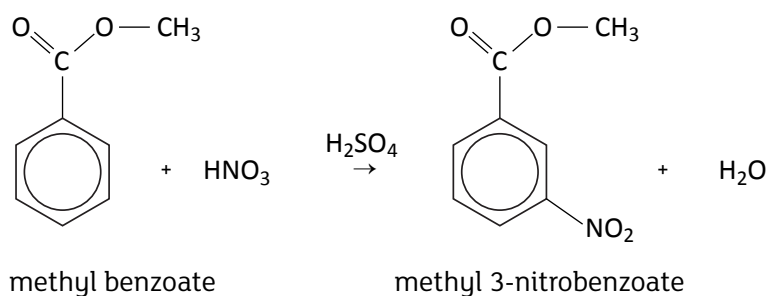


Practical 6.1

Preparation of methyl 3-nitrobenzoate (spec ref 4.10.5)

Introduction

Benzene and other compounds containing a benzene ring may be nitrated using a mixture of concentrated sulfuric acid and concentrated nitric acid. This 'nitrating mixture' forms the nitronium ion, NO_2^+ , which acts as an electrophile and reacts with the benzene ring in a substitution reaction. In this practical you nitrate the benzene ring present in methyl benzoate, a safer alternative to benzene itself:



The reaction must be performed at a low temperature to prevent further nitration of the benzene ring.

Apparatus and materials

- safety glasses & protective gloves
- 2 × 100 cm³ conical flasks
- 3 × 10 cm³ measuring cylinders
- test tube
- plastic dropping pipette
- thermometer (-10 – 100 °C)
- 250 cm³ beaker
- glass rod
- Büchner funnel and flask
- filter paper
- watch glass
- access to a drying oven
- access to a mass balance (2 d.p)
- melting point apparatus
- a capillary tube sealed at one end
- concentrated sulfuric acid



Practical 6.1

Preparation of methyl 3-nitrobenzoate (spec ref 4.10.5)

- methyl benzoate
- concentrated nitric acid
- ethanol
- ice
- deionised water

Procedure

1. Measure 9 cm³ of concentrated sulfuric acid using a measuring cylinder and transfer into a 100 cm³ conical flask. Place the flask in an iced water bath.
2. Add 4 cm³ of methyl benzoate to a measuring cylinder. Weigh the measuring cylinder and methyl benzoate and record the mass in the table below.
3. Measure 3 cm³ of concentrated nitric acid using a measuring cylinder and transfer to a test tube. Measure 3 cm³ of concentrated sulfuric acid using a measuring cylinder and transfer to the test tube. Place the test tube in the iced water bath.
4. Add the methyl benzoate to the conical flask while gently swirling the flask. Reweigh the empty measuring cylinder and record the mass in the table.
5. Place a thermometer in the conical flask. When the temperature reaches between 0–10 °C, use a plastic dropping pipette to add the nitrating mixture prepared in step 3 drop wise from the test tube to the conical flask, ensuring the temperature does not rise above 10 °C.
6. When the addition is complete, remove the conical flask from the iced water bath and allow to stand at room temperature for 5 minutes. Pour the reaction mixture from flask into a beaker containing crushed ice and water. Stir until the product solidifies.
7. Filter off the solid by suction filtration using a Büchner funnel and flask. Wash the solid product first with two portions of deionised water then with a 5 cm³ of cold ethanol.
8. Transfer the solid to a 100 cm³ conical flask. Add 10 cm³ ethanol then heat the mixture in a water bath. If the solid fails to dissolve add further 2 cm³ portions of ethanol until a solution is obtained. Cool the flask in an iced water bath until crystals form.
9. Filter off the crystals by suction filtration. Wash with 5 cm³ of cold ethanol and then transfer the crystals to a watch glass. Dry the crystals in a drying oven overnight.
10. Weigh the crystals and record their mass in the table.
11. Insert some of the crystals into a capillary tube sealed at one end and then insert into the melting point apparatus.
12. Slowly heat the sample and observe the solid in the capillary tube. Note the temperature when the solid starts and stops melting.



Practical 6.1

Preparation of methyl 3-nitrobenzoate (spec ref 4.10.5)

Mass of measuring cylinder and methyl benzoate / g	
Mass of emptied measuring cylinder / g	
Mass of methyl benzoate / g	
Mass of methyl 3-nitrobenzoate / g	
Melting point of methyl 3-nitrobenzoate / °C	

Questions

1. Calculate the percentage yield of methyl 3-nitrobenzoate. Suggest reasons why the yield is less than 100%.

2. Over what temperature range did the crystals melt? How does this indicate the purity of the sample?



Practical 6.1

Preparation of methyl 3-nitrobenzoate (spec ref 4.10.5)

3. Write an equation for the reaction between the concentrated nitric acid and concentrated sulfuric acid to produce the nitronium ion.

4. Draw a flow scheme for the reaction between the nitronium ion and methyl benzoate.

5. Explain why the temperature is controlled during the addition of the nitrating mixture to methyl benzoate.
