AH Chem Practicals

1A

Preparation of a Standard Solution

Introduction

A standard solution is one of accurately known concentration and can be prepared directly from a primary standard which, in this case, is hydrated oxalic acid, $(COOH)_2 \bullet 2H_2O$ (RFM = 126.1).
To prepare 250 cm^3 of 0.1 mol l^{-1} oxalic acid solution, the mass of hydrated oxalic acid required can be calculated as 0.1 × 0.250 × 126.1 = 3.15 g.

Requirements

balance (accurate to 0.01g)
oxalic acid AnalaR, $(COOH)_2 \bullet 2H_2O$
weighing bottle
deionised water
250cm^3 beaker
250cm^3 standard flask
wash bottle
dropper
glass stirring rod
filter funnel

Hazcon

Wear eye protection and if any chemical splashes on the skin, wash it off immediately.
Oxalic acid is harmful if ingested and irritates the eyes and skin. Wear gloves.

Procedure

1. Transfer approximately 3.2 g of oxalic acid crystals to the weighing bottle and weigh accurately.
2. Pour the oxalic acid crystals into a clean beaker containing about 50cm^3 of deionised water and reweigh accurately the weighing bottle and any remaining crystals.
3. Stir the solution until all the oxalic acid dissolves and then transfer it to a 250cm^3 standard flask.
4. Rinse the beaker several times with deionised water and add all the rinsings to the flask.
5. Make up the solution to the graduation mark with deionised water.
6. Stopper the flask and invert it several times to ensure the contents are completely mixed.
7. Calculate the concentration of the oxalic acid solution using the exact mass of the oxalic acid transferred to the beaker in step 2.

AH Chem Practicals

1B

Standardisation of Sodium Hydroxide

Introduction

Sodium hydroxide is not a primary standard and so a standard solution of it cannot be prepared directly from the solid. However, a solution of approximate concentration can be prepared and its exact concentration determined by titrating it against an acid of accurately known concentration using a suitable indicator. In this experiment, a sodium hydroxide solution is standardised against the 0.1 mol l^{-1} oxalic acid solution prepared in Experiment 1A. The stoichiometric equation for the titration reaction is:

$$(COOH)_2 + 2NaOH \longrightarrow 2H_2O + (COO^-Na^+)_2$$

Requirements

standardised oxalic acid solution (approx. 0.1 mol l^{-1})
sodium hydroxide solution (approx. 0.1 mol l^{-1})
phenolphthalein indicator
deionised water
100cm^3 beakers
100cm^3 conical flasks
wash bottle
pipette filler
white tile
filter funnel
50cm^3 burette
10cm^3 pipette

Hazcon

Wear eye protection and if any chemical splashes on the skin, wash it off immediately.
0.1 mol l^{-1} oxalic acid irritates the eyes and skin.
0.1 mol l^{-1} sodium hydroxide is corrosive to the eyes and skin.
Phenolphthalein indicator solution is highly flammable and irritating to the eyes because of its ethanol content.

Procedure

1. Rinse the 10cm^3 pipette with a little of the oxalic acid solution and pipette 10cm^3 of it into a conical flask.
2. Add two or three drops of phenolphthalein indicator to the oxalic acid solution in the flask.
3. Rinse the 50cm^3 burette, including the tip, with the sodium hydroxide solution and fill it with the same solution.
4. Titrate the oxalic acid solution with the sodium hydroxide solution from the burette until the end-point is reached. This is indicated by the appearance of a pink colour.
5. Repeat the titrations until two concordant results are obtained.
6. Calculate the concentration of the sodium hydroxide solution.

AH Chem Practicals

1C

Determination of Ethanoic Acid in Vinegar

Introduction

Vinegar is a dilute solution of ethanoic acid and the aim of this experiment is to determine the concentration of ethanoic acid in a given sample of white vinegar by titration against the sodium hydroxide solution standardised in Experiment 1B. The stoichiometric equation for the titration reaction is:

$$CH_3COOH + NaOH \longrightarrow H_2O + CH_3COONa$$

Requirements

white vinegar
standardised sodium hydroxide solution (approx. 0.1 mol l^{-1})
deionised water
phenolphthalein indicator
250cm^3 standard flask
100cm^3 conical flasks
100cm^3 beakers
wash bottle
pipette filler
dropper
white tile
filter funnel
50cm^3 burette
25cm^3 pipette

Hazcon

Wear eye protection and if any chemical splashes on the skin, wash it off immediately.
Vinegar irritates the eyes and skin.
0.1 mol l^{-1} sodium hydroxide is corrosive to the eyes and skin.
Phenolphthalein indicator solution is highly flammable and irritating to the eyes because of its ethanol content.

Procedure

1. Rinse the 25cm^3 pipette with a little of the vinegar.
2. Dilute the sample of vinegar by pipetting 25cm^3 of it into a clean 250cm^3 standard flask and making it up to the graduation mark with deionised water.
3. Stopper the standard flask and invert it several times to ensure the contents are thoroughly mixed.
4. Rinse the 25cm^3 pipette with a little of the diluted vinegar and pipette 25cm^3 of it into a conical flask.

AH Chem Practicals

2A

Preparation of Standard Solution

Introduction

A standard solution is one of accurately known concentration and can be prepared directly from a primary standard which, in this case, is anhydrous sodium carbonate, Na_2CO_3 (RFM = 106.0).
To prepare 250cm^3 of 0.1mol l^{-1} sodium carbonate solution, the mass of anhydrous sodium carbonate required can be calculated as 0.1 × 0.250 × 106.0 = 2.65g.

Requirements

balance (accurate to 0.01g)
anhydrous sodium carbonate AnalaR
evaporating basin
deionised water
desiccator
weighing bottle
Bunsen burner, heating mat and tripod
250cm^3 beaker
250cm^3 standard flask
wash bottle
dropper
glass stirring rod
filter funnel

Hazcon

Wear eye protection and if any chemical splashes on the skin, wash it off immediately.
Sodium carbonate powder is harmful if inhaled and irritates the eyes.

Procedure

1. Heat gently and with constant stirring, approximately 10g of anhydrous sodium carbonate in an evaporating basin, for about 15 minutes.
2. Place the evaporating basin and contents in a desiccator.
3. After cooling, weigh the evaporating basin and contents.
4. Heat the sodium carbonate again for about 5 minutes, allow to cool in the desiccator and reweigh. Repeat this process until the mass is constant.
5. Transfer approximately 2.65g of the dried anhydrous sodium carbonate to the weighing bottle and weigh accurately.
6. Add the anhydrous sodium carbonate to a clean beaker containing about 50cm^3 of deionised water and reweigh accurately the weighing bottle and any remaining powder.
7. Stir the solution until all the sodium carbonate dissolves and then transfer it to a 250cm^3 standard flask.
8. Rinse the beaker several times with deionised water and add all the rinsings to the flask.
9. Make up the solution to the graduation mark with deionised water.
10. Stopper the flask and invert it several times to ensure the contents are completely mixed.
11. Calculate the concentration of the sodium carbonate solution using the exact mass of the anhydrous sodium carbonate transferred to the beaker in step 6.

AH Chem Practicals

2B

Standardisation of Hydrochloric Acid

Introduction

Hydrochloric acid is not a primary standard and so a standard solution of it cannot be prepared directly. However, a solution of approximate concentration can be prepared and its exact concentration determined by titrating it against a base of accurately known concentration using a suitable indicator. In this experiment, approximately 1mol l^{-1} hydrochloric acid is first diluted and then standardised against the 0.1mol l^{-1} sodium carbonate solution prepared in Experiment 2A. The stoichiometric equation for the titration reaction is:

$$Na_2CO_3 + 2HCl \longrightarrow H_2O + CO_2 + 2NaCl$$

Requirements

standardised sodium carbonate solution (approx. 0.1mol l^{-1})
hydrochloric acid (approx. 1mol l^{-1})
screened methyl orange indicator (or any other suitable indicator)
100cm^3 beakers
250cm^3 standard flask
100cm^3 conical flasks
50cm^3 burette
10cm^3 and 25cm^3 pipettes
pipette filler
deionised water
wash bottle
dropper
white tile
filter funnel

Hazcon

Wear eye protection and if any chemical splashes on the skin, wash it off immediately.
1mol l^{-1} hydrochloric acid irritates the eyes and skin.

Procedure

1. Rinse the 25cm^3 pipette with a little of the 1 mol l^{-1} hydrochloric acid solution.
2. Dilute the sample of hydrochloric acid by pipetting 25cm^3 of it into a clean 250cm^3 standard flask and making it up to the graduation mark with deionised water.
3. Stopper the standard flask and invert it several times to ensure the contents are thoroughly mixed.
4. Rinse the 10cm^3 pipette with a little of the sodium carbonate solution and pipette 10cm^3 of it into a conical flask.
5. Add two or three drops of screened methyl orange indicator to the sodium carbonate solution in the flask.
6. Rinse the 50cm^3 burette, including the tip, with the diluted hydrochloric acid and fill it with the same solution.
7. Titrate the sodium carbonate solution with the diluted hydrochloric acid from the burette until the end-point is reached. This is indicated by a green to mauve colour change.
8. Repeat the titrations until two concordant results are obtained.
9. Calculate the concentration of the diluted hydrochloric acid and hence the undiluted hydrochloric acid.

AH Chem Practicals

2C

Determination of Purity of Marble

Introduction

Marble (calcium carbonate) is insoluble in water and so the calcium carbonate content has to be determined by a back titration technique. This involves treating a sample of marble of accurately known mass with a definite amount of hydrochloric acid, ie the volume and concentration of the acid sample must be known accurately. An excess of acid is used and the amount remaining after neutralising the calcium carbonate is determined by titrating it against a standard solution of sodium hydroxide.

Requirements

standardised 1.0mol l^{-1} hydrochloric acid
standardised 0.1mol l^{-1} sodium hydroxide
screened methyl orange indicator (or any other suitable indicator)
$25cm^3$ pipette
$50cm^3$ pipette
weighing bottle
balance (accurate to 0.01 g)
wash bottle
filter funnel
$250cm^3$ standard flask
$100cm^3$ glass beakers
$100cm^3$ conical flasks
$50cm^3$ burette
marble chips
pipette filler
dropper
white tile
deionised water

Hazcon

Wear eye protection and if any chemical splashes on the skin, wash it off immediately.
Both 1.0mol l^{-1} hydrochloric acid and 0.1mol l^{-1} sodium hydroxide irritate the eyes and skin.

Procedure

1. Transfer approximately 1.0g of marble chips to a weighing bottle and weigh the bottle and contents.
2. Transfer the marble chips to the $250cm^3$ standard flask and reweigh the bottle.
3. Rinse the $50cm^3$ pipette with a little of the 1mol l^{-1} hydrochloric acid and pipette $50cm^3$ of it into the standard flask.
4. When effervescence has stopped, make up the solution in the flask to the graduation mark with deionised water.
5. Stopper the standard flask and invert it several times to ensure the contents are thoroughly mixed.
6. Rinse the $50cm^3$ burette, including the tip, with the 0.1mol l^{-1} sodium hydroxide solution and fill it.
7. Rinse the $25cm^3$ pipette with some of the 'standard flask' solution and pipette $25cm^3$ of this solution into a conical flask.
8. Add two or three drops of screened methyl orange indicator to the solution in the flask.
9. Titrate the 'standard flask' solution with the sodium hydroxide solution from the burette until the end-point is reached. This is indicated by a mauve to green colour change.
10. Repeat the titrations until two concordant results are obtained.
11. Calculate the percentage by mass of calcium carbonate in the marble sample using the accurate concentrations of the hydrochloric acid and sodium hydroxide solutions provided by your practitioner.

AH Chem Practicals
3

Determination of Nickel using EDTA

Introduction

Since EDTA forms stable complexes with most metal ions, it is widely used to determine metals in what are known as complexometric titrations. EDTA is a tetracarboxylic acid and can be represented as H_4Y. In alkaline conditions, it exists as Y^{4-} ions, which form 1:1 complexes with metal ions such as nickel(II) ions:

$$Y^{4-} + Ni^{2+} \longrightarrow NiY^{2-}$$

The end-point of an EDTA complexometric titration can be detected by means of a metal ion indicator – an organic dye which changes colour when it binds with metal ions. For it to be suitable in an EDTA titration, the indicator must bind less strongly with metal ions than does EDTA. Murexide is one such indicator.

Requirements

hydrated nickel(II) sulfate ($NiSO_4 \bullet 6H_2O$)
standardised 0.10mol l^{-1} EDTA solution
1mol l^{-1} ammonium chloride
murexide indicator
0.88 aqueous ammonia
balance (accurate to 0.01 g)
wash bottle
pipette filler
white tile
deionised water

$50cm^3$ burette
$20cm^3$ pipette
$100cm^3$ standard flask
$250cm^3$ conical flasks
$100cm^3$ beakers
$25cm^3$ measuring cylinder
weighing bottle
filter funnel
glass stirring rod

Hazcon

Wear eye protection and if any chemical splashes on the skin, wash it off immediately.
Hydrated nickel(II) sulfate is harmful by ingestion and inhalation. Wear gloves.
EDTA is only toxic if ingested in large quantities.
0.88 aqueous ammonia is toxic if inhaled in high concentrations or if swallowed. The solution and vapour irritate the eyes. The solution burns the skin. Wear goggles and gloves and handle it in a fume cupboard.
1 mol l^{-1} ammonium chloride is harmful and irritates the eyes.
Murexide is harmful by ingestion and if inhaled as a dust.

Procedure

1. Transfer approximately 2.6g of hydrated nickel(II) sulfate to a weighing bottle and weigh the bottle and contents.
2. Add about 25cm^3 of deionised water to a 100cm^3 beaker and transfer the bulk of the nickel salt to the water.
3. Reweigh the bottle with any remaining salt.
4. Stir the mixture until the solid dissolves and transfer the resulting solution to a 100cm^3 standard flask.
5. Rinse the beaker several times with a little deionised water and add the rinsings to the standard flask.
6. Make up the solution to the graduation mark with deionised water. Stopper the flask and invert it several times to ensure the contents are thoroughly mixed.
7. Rinse the burette, including the tip, with 0.01 mol l^{-1} EDTA and fill it with the same solution.
8. Rinse the 20 cm^3 pipette with a little of the nickel salt solution and pipette 20cm^3 of it into a conical flask. Dilute the solution to about 100cm^3 with deionised water.
9. Add murexide indicator (approximately 0.05g) to the diluted nickel salt solution together with approximately 10cm^3 of ammonium chloride solution.
10. Titrate the mixture with the EDTA solution and after the addition of about 15cm^3 make the solution alkaline by adding approximately 10cm^3 of 0.88 aqueous ammonia (concentrated ammonia solution).
11. Continue the titration to the end-point, which is shown by the first appearance of a blue-violet colour. Detection of the end-point can be difficult so keep this titrated solution to help you detect end-points in subsequent titrations.
12. Repeat the titrations until two concordant results are obtained.
13. Calculate the percentage by mass of nickel in the sample of hydrated nickel(II) sulfate using the accurate concentration of the EDTA solution provided by your practitioner.
14. Calculate the theoretical percentage by mass of nickel in $NiSO_4 \bullet 6H_2O$ and compare this with the experimental value. Account for any difference.

AH Chem Practicals

4A

Water in Hydrated Barium Chloride

Introduction

Gravimetric analysis can be used to determine the number of moles of water molecules of crystallisation per mole of hydrated barium chloride, ie the value of *n* in $BaCl_2.nH_2O$. This can be achieved by comparing the mass of a sample of the hydrated salt with the mass of the anhydrous salt obtained on heating to constant mass.

Requirements

hydrated barium chloride
silica or porcelain crucible and lid
tripod
pipe-clay triangle
Bunsen burner and heating mat
desiccator
tongs
balance (preferably accurate to 0.001 g)

Hazcon

Wear eye protection and if any chemical splashes on the skin, wash it off immediately.
Barium chloride is harmful by inhalation and by ingestion or skin contact. Wear gloves.

Procedure

1. Place the empty crucible and lid on the pipe-clay triangle and heat them for about 10 minutes using a blue Bunsen flame. Heating should be gentle at first.
2. Allow the crucible and lid to cool briefly before transferring them, using clean tongs, to the desiccator.
3. After cooling to room temperature, weigh the empty crucible and lid.
4. Add 2–3g of hydrated barium chloride to the crucible. Replace the lid and reweigh.
5. Place the crucible back on the pipe-clay triangle with the lid partially covering the contents. Heat gently for about 2 minutes and then strongly for 10–15 minutes.
6. Allow the crucible to cool briefly before transferring it to the desiccator.
7. Once they have cooled to room temperature, reweigh the crucible and contents.

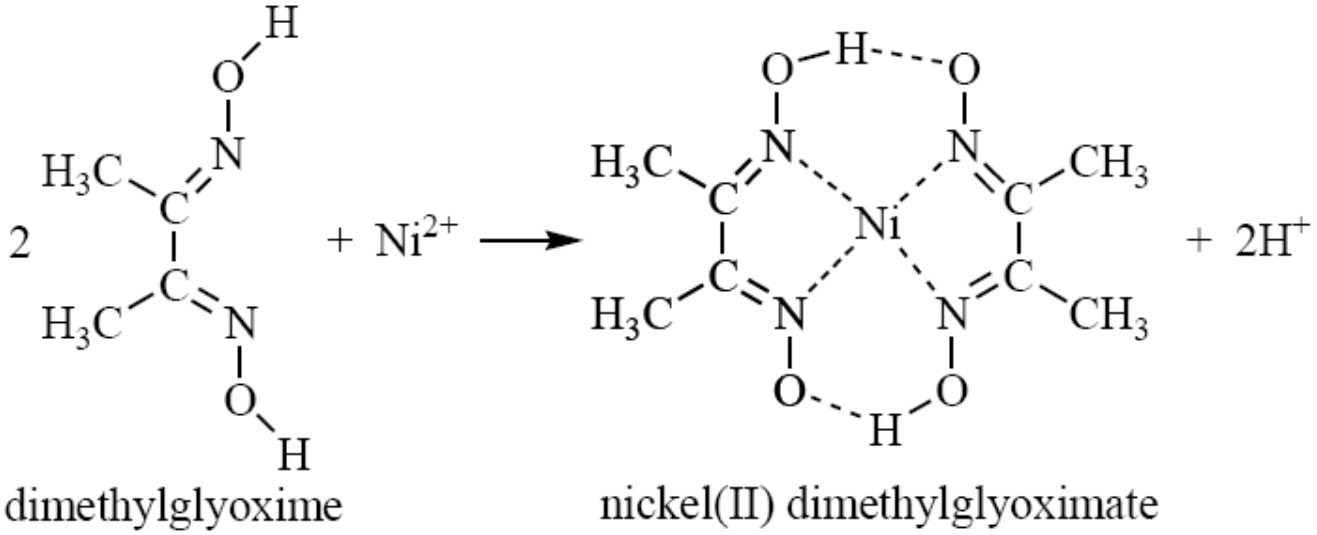
AH Chem Practicals

4B

Determination of Nickel Using Dimethylglyoxime

Introduction

Gravimetric analysis can be used to determine the nickel content of a nickel(II) salt. This can be achieved by reacting the nickel(II) ions with dimethylglyoxime (butanedione dioxime) in the presence of a slight excess of ammonia:



The complex, nickel(II) dimethylglyoximate, is filtered from the reaction mixture, dried and weighed.

Requirements

hydrated nickel(II) chloride ($NiCl_2 \bullet 6H_2O$)
2mol l^{-1} ammonia
0.1mol l^{-1} dimethylglyoxime in ethanol
2mol l^{-1} hydrochloric acid
desiccator
balance (preferably accurate to 0.001 g)
weighing bottle
hot plate
steam bath
500 cm^3 beaker
sintered glass crucible
Buchner flask and adapter
water pump
measuring cylinders (10cm^3 and 100cm^3)
thermometer
stirring rod
dropper
oven

Hazcon

Wear eye protection and if any chemical splashes on the skin, wash it off immediately.
Hydrated nickel(II) chloride is harmful by inhalation and by ingestion. Wear gloves.
Dimethylglyoxime in ethanol is irritating to the eyes and is highly flammable.
2 mol l^{-1} ammonia irritates the eyes.

Procedure

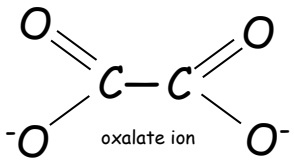
1. Transfer approximately 0.5g of hydrated nickel(II) chloride to a weighing bottle and weigh the bottle and contents.
2. Add about 20cm^3 of deionised water to a 500cm^3 beaker and transfer the bulk of the nickel salt to the water.
3. Reweigh the bottle with any remaining salt.
4. Stir the mixture until the solid dissolves and add about 20cm^3 of 2mol l^{-1} hydrochloric acid. Dilute the mixture with deionised water to about 200cm^3.
5. Heat the solution to 70–80°C on a hot plate and add approximately 50cm^3 of 0.1mol l^{-1} dimethylglyoxime in ethanol.
6. Add 2mol l^{-1} ammonia solution dropwise and with constant stirring until a permanent red precipitate is obtained. Add a further 5cm^3 of the ammonia solution to provide a slight excess. In all, you should have added about 30cm^3 of ammonia solution.
7. Heat the beaker and contents on a steam bath for about 30 minutes and when the precipitate has settled test the clear liquid for complete precipitation by adding a few drops of the dimethylglyoxime and ammonia solutions. (If more red precipitate appears then add about 5cm^3 of 0.1 mol l^{-1} dimethylglyoxime solution followed by about 3cm^3 of 2mol l^{-1} ammonia solution.)
8. Remove the beaker from the steam bath and allow it to cool to room temperature.
9. Dry the sintered glass crucible in an oven at 120°C, allow it to cool in a desiccator and then weigh it.
10. Set up the filtration apparatus: sintered glass crucible, Buchner flask and adapter. Filter off the precipitate at the water pump and wash the precipitate with a several portions of deionised water.
11. Dry the crucible and precipitate in the oven at 120°C for about 1 hour and then transfer them to a desiccator.
12. Once they have cooled to room temperature, reweigh the crucible and contents.
13. Heat the crucible and contents to constant mass, ie reheat for about 15 minutes in the oven at 120°C, cool in the desiccator and reweigh until two successive readings are within 0.002g of each other or within 0.01g of each other if the balance available is only accurate to 0.01g.
14. Calculate the percentage by mass of nickel in the sample of the hydrated nickel(II) chloride.
15. Calculate the theoretical percentage by mass of nickel in $NiCl_2 \bullet 6H_2O$ and compare this with the experimental value. Account for any difference.

AH Chem Practicals

5

Preparation of Potassium Trioxalatoferrate (III)

Introduction



oxalate ion

Potassium trioxalatoferrate(III) contains the complex ion, $[Fe(C_2O_4)_3]^{3-}$, in which three oxalate ions bind to an iron(III) ion in an octahedral arrangement. The oxalate ions behave as ligands.

Potassium trioxalatoferrate(III) can be prepared from ammonium iron(II) sulfate. A solution of the latter is first treated with oxalic acid to form a precipitate of iron(II) oxalate and ammonium hydrogensulfate solution.

$$\underset{\text{ammonium iron(II) sulfate}}{(NH_4)_2Fe(SO_4)_2} + \underset{\text{oxalic acid}}{H_2C_2O_4} \longrightarrow \underset{\text{iron oxalate}}{FeC_2O_4} + \underset{\text{ammonium hydrogensulfate}}{2NH_4HSO_4}$$

The iron(II) oxalate is isolated from the mixture and on reaction with hydrogen peroxide and potassium oxalate, potassium trioxalatoferrate(III) and a precipitate of iron(III) hydroxide are produced.

$$\underset{\text{iron oxalate}}{6FeC_2O_4} + \underset{\text{hydrogen peroxide}}{3H_2O_2} + \underset{\text{potassium oxalate}}{6K_2C_2O_4} \longrightarrow \underset{\text{potassium trioxalatoferrate(III)}}{4K_3[Fe(C_2O_4)_3]} + \underset{\text{iron (III) hydroxide}}{2Fe(OH)_3}$$

On further treatment with oxalic acid, the iron(III) hydroxide reacts to form more potassium trioxalatoferrate(III):

$$\underset{\text{iron (III) hydroxide}}{2Fe(OH)_3} + \underset{\text{oxalic acid}}{3H_2C_2O_4} + \underset{\text{potassium oxalate}}{3K_2C_2O_4} \longrightarrow \underset{\text{potassium trioxalatoferrate(III)}}{2K_3[Fe(C_2O_4)_3]} + \underset{\text{water}}{6H_2O}$$

On cooling, crystals of hydrated potassium trioxalatoferrate(III), $K_3[Fe(C_2O_4)_3].3H_2O$, separate from the reaction mixture.

Requirements

hydrated ammonium iron(II) sulfate ($(NH_4)_2Fe(SO_4)_2 \bullet 6H_2O$)
oxalic acid solution (100 g l^{-1})
potassium oxalate solution (300g l^{-1})
dilute sulfuric acid (2mol l^{-1})
'20 volume' hydrogen peroxide
deionised water
ethanol
filter papers
100 cm^3 crystallising basin
100cm^3 glass beakers
balance (accurate to 0.01g)
hot plate
glass stirring rod
25 cm^3 measuring cylinder
thermometer
dropper
glass filter funnel
clock glass

Hazcon

Wear eye protection and if any chemical splashes on the skin, wash it off immediately.
Hydrated ammonium iron(II) sulfate may be harmful if ingested and may irritate the eyes. Wear gloves.
Oxalic acid solution, potassium oxalate solution and the product, potassium trioxalatoferrate(III), are all harmful by ingestion and are irritating to the eyes and skin. Wear gloves.
'20 volume' hydrogen peroxide is irritating to the eyes and skin. Wear gloves.
Ethanol is volatile, highly flammable, irritating to the eyes and intoxicating if inhaled or ingested.
Dilute sulfuric acid is corrosive. Wear gloves.

Procedure

1. Weigh a 100 cm^3 glass beaker and to it add approximately 5g of hydrated ammonium iron(II) sulfate, $(NH_4)_2Fe(SO_4)_2.6H_2O$. Reweigh the beaker and its contents.
2. Add approximately 15cm^3 of deionised water and 1cm^3 of dilute sulfuric acid to the ammonium iron(II) sulfate. Warm the mixture to dissolve the solid.
3. Once the ammonium iron(II) sulfate has dissolved, add 25cm^3 of oxalic acid solution.
4. Place the beaker on a hot plate and slowly heat the mixture with stirring until it boils.
5. Remove the beaker from the heat and allow the precipitate of iron(II) oxalate to settle to the bottom of the beaker.
6. Decant off the liquid and add about 50cm^3 of hot deionised water to the precipitate. Stir the mixture and after the precipitate has settled once more, decant off the liquid.
7. Add 10cm^3 of potassium oxalate solution to the washed precipitate and heat the mixture to about 40°C.
8. To this mixture, add slowly with continuous stirring 20cm^3 of '20 volume' hydrogen peroxide. Keep the temperature close to 40°C during the addition of the hydrogen peroxide.
9. Heat the mixture nearly to boiling and add oxalic acid solution, dropwise with stirring, until the brown precipitate of iron(III) hydroxide dissolves. Take care not to add too much oxalic acid. During the addition of the oxalic acid, keep the reaction mixture near to boiling.
10. Filter the hot solution through a fluted filter paper into a crystallising basin.
11. Add 25cm^3 of ethanol to the filtrate and if any crystals form, redissolve them by gentle heating.
12. Cover the solution with a filter paper and set it aside in a dark cupboard for crystallisation to take place.
13. Filter off the crystals and wash them with a 1:1 mixture of ethanol and water.
14. Weigh a clock glass and transfer the crystals to it. Cover the crystals with a filter paper and leave them to dry at room temperature in a dark cupboard.
15. Once dry, reweigh the crystals and clock glass.
16. Calculate the percentage yield of hydrated potassium trioxalatoferrate(III), $K_3[Fe(C_2O_4)_3]\bullet 3H_2O$.

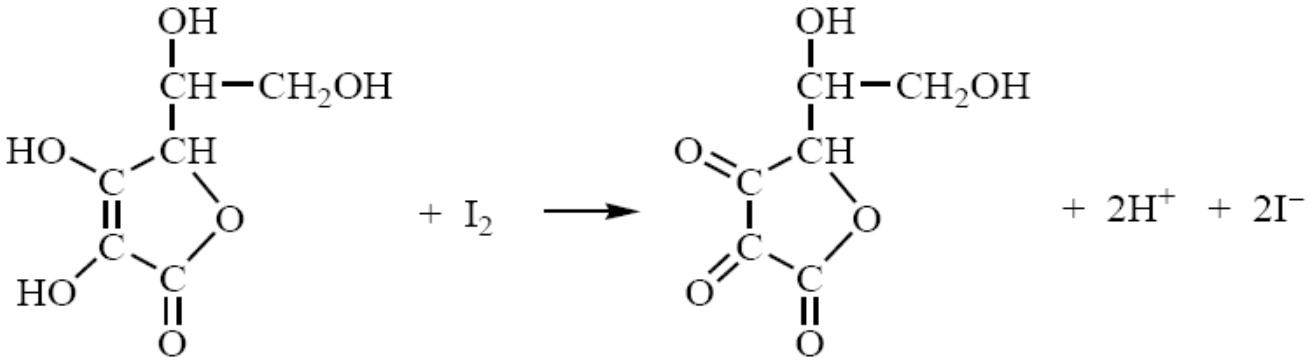
AH Chem Practicals

6

Determination of Vitamin C

Introduction

Vitamin C (ascorbic acid) is an important component of our diet. Although it occurs naturally in many fruits and vegetables, many people take vitamin C tablets to supplement their intake. The vitamin C content of a tablet can be determined by carrying out a redox titration with a standard solution of iodine using starch solution as indicator:



vitamin C
(ascorbic acid)

It is good practice, especially when using an unfamiliar procedure, to carry out a control experiment. In this case the control would involve carrying out the determination of vitamin C (ascorbic acid) using a pure sample of the compound. If the mass of vitamin C (ascorbic acid) you determine matches the mass you started with then this establishes the validity of the procedure and the results. However, if the experimental result deviates significantly from the true value then this could arise from bad technique or not using standardised solutions. These should be checked before dismissing a procedure as invalid.

Requirements

1g effervescent vitamin C tablet
sample of pure ascorbic acid
standardised 0.025mol l^{-1} iodine solution
starch solution
deionised water
balance (accurate to 0.01g)
pipette filler
filter funnel
100cm^3 beakers
250cm^3 standard flask
100cm^3 conical flasks
25cm^3 pipette
50cm^3 burette
weighing bottle
dropper
white tile
wash bottle

Hazcon

Wear eye protection and if any chemical splashes on the skin, wash it off immediately.
0.025 mol l^{-1} iodine solution irritates the eyes and causes severe internal irritation if swallowed. Wear gloves and treat any spills on the skin with sodium thiosulfate solution.

Procedure

Control experiment using pure ascorbic acid

1. Add about 1.0g of pure ascorbic acid to the weighing bottle and weigh the bottle and contents.
2. Transfer the pure ascorbic acid to a beaker and reweigh the weighing bottle.
3. Add some deionised water (approximately 50cm^3) to the beaker and stir the mixture until the ascorbic acid dissolves.
4. Transfer the solution to a 250cm^3 standard flask.
5. Rinse the beaker with a little deionised water and add the rinsings to the standard flask. Repeat this procedure several times and add the rinsings to the flask. Make up the solution to the graduation mark with deionised water.
6. Stopper the flask and invert it several times to ensure the contents are completely mixed.
7. Rinse the burette, including the tip, with 0.025mol l^{-1} iodine solution and fill it with the same solution.
8. Rinse the 25cm^3 pipette with the ascorbic acid solution and pipette 25cm^3 of it into a 100cm^3 conical flask.
9. Add a few drops of starch indicator to the solution and titrate to the end-point, which is indicated by the colour changing to blue.
10. Repeat the titrations until two concordant results are obtained.
11. Calculate the mass of ascorbic acid in the initial sample using the accurate concentration of the iodine solution provided by your practitioner.
12. Compare your result with the initial mass of pure ascorbic acid you used.

Determination of vitamin C (ascorbic acid) in a commercial tablet

1. Add a 1g effervescent vitamin C tablet to a beaker.
2. Repeat steps 2 to 10 of the above procedure.
3. Calculate the mass of vitamin C in the tablet using the accurate concentration of the iodine solution provided by your practitioner.
4. Compare your result with the manufacturer's specification.

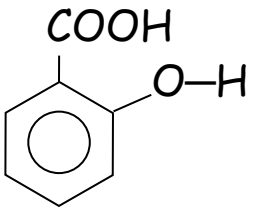
AH Chem Practicals

7A

Preparation of Aspirin

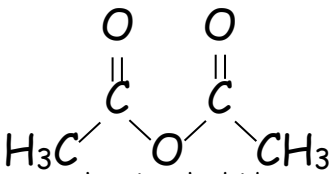
Introduction

Aspirin (acetyl salicylic acid) is an analgesic (pain-killing), anti-inflammatory and antipyretic (fever-reducing) drug. It is an ester and can be prepared by the condensation reaction between 2-hydroxybenzoic acid (salicylic acid) and ethanoic anhydride:



2-hydroxybenzoic acid

After purification by recrystallisation, the product can be weighed and the percentage yield determined. The purity and identity of the final sample can be checked by measuring its melting point and mixed melting point, and by thin-layer chromatography.



ethanoic anhydride

Requirements

2-hydroxybenzoic acid
85% phosphoric acid
ethanoic anhydride
ethanol
anti-bumping granules
deionised water
ice
sample of pure aspirin
iodine
dichloromethane
ethyl ethanoate
filter papers
clock glass
oven
capillary tubes
melting point apparatus

$50cm^3$ conical flask
$100cm^3$ conical flasks
measuring cylinders ($10cm^3$ and $50cm^3$)
$250cm^3$ glass beakers
thermometers
dropper
glass stirring rod
balance (accurate to 0.01g)
hot plate
Buchner funnel and flask
water pump
chromatography chamber
TLC plate
test-tubes
UV lamp

Hazcon

Wear eye protection and if any chemical splashes on the skin, wash it off immediately.
2-Hydroxybenzoic acid is harmful by ingestion, causing nausea, vomiting etc. It is also a severe skin and eye irritant. Wear gloves.
Ethanoic anhydride is corrosive. The liquid irritates and burns the eyes and skin severely while the vapour irritates the respiratory system and may cause bronchial and lung injury. It is also flammable. Wear gloves and handle in a fume cupboard.
85% phosphoric acid is corrosive: it burns and irritates the skin and eyes. It is a systemic irritant if inhaled and if swallowed causes serious internal injury. Wear gloves.
Aspirin irritates the eyes and skin.
Ethanol is volatile, highly flammable, irritating to the eyes and intoxicating if inhaled or ingested.
Dichloromethane irritates the eyes and skin and is at its most harmful if inhaled. Wear gloves.
Ethyl ethanoate is irritating to the eyes, volatile and can irritate the respiratory system. It is highly flammable. Wear gloves.

Procedure

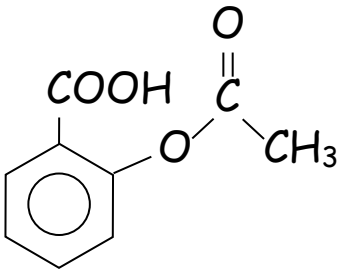
1. Weigh a 50cm^3 conical flask and to it add about 5g of 2-hydroxybenzoic acid. Reweigh the flask and its contents.
2. In a fume cupboard, add 10cm^3 of ethanoic anhydride from a measuring cylinder to the 2-hydroxybenzoic acid. During the addition, swirl the contents of the flask to ensure thorough mixing.
3. Add five drops of 85% phosphoric acid to the mixture, again with swirling.
4. Place the flask on a hot plate (in the fume cupboard) and heat the mixture to about 85°C. Keep it at this temperature for about 10 minutes and constantly stir the mixture.
5. Cool the mixture in an ice/water bath and then pour it into approximately 150cm^3 of cold water contained in a 250cm^3 beaker.
6. Filter off the precipitate at the water pump and wash it thoroughly with several portions of cold water.
7. Transfer the crude product to about 15cm^3 of ethanol in a 100cm^3 conical flask. Add a couple of anti-bumping granules and heat mixture gently on a hot plate until it dissolves.
8. Pour this solution into a 100cm^3 conical flask containing about 40cm^3 of water. If an oil forms, reheat the mixture on a hot plate to dissolve it. If the oil persists, add a few drops of ethanol and reheat the mixture.
9. Set aside the mixture and allow it to cool to room temperature.
10. Filter off the crystals of aspirin at the water pump and wash them with a small volume of cold water. Allow air to be drawn through the crystals for a few minutes in order to partially dry them.
11. Weigh a clock glass and transfer the crystals to it. Dry the crystals in an oven at about 100°C and then reweigh the clock glass and crystals.
12. Calculate the percentage yield of aspirin.
13. Determine the melting point of the aspirin product.
14. Grind a 50:50 mixture of product and pure sample of aspirin and determine the mixed melting point. This gives an indication of the purity of the aspirin you have prepared.
15. Take a TLC plate and using a pencil lightly draw a line across the plate about 1 cm from the bottom. Mark two well-spaced points on the line.
16. Place small amounts (about a third of a spatulaful) of your aspirin product and a pure sample of aspirin in two separate test-tubes.
17. Add about 1cm^3 of solvent (a 50:50 mixture of ethanol and dichloromethane) to each of the test-tubes to dissolve the aspirin samples.
18. Use capillary tubes to spot each of the two samples onto the TLC plate. Allow to dry and repeat two or three more times.
19. After the spots have dried, place the TLC plate into the chromatography chamber, making sure that the pencil line is above the level of the solvent (ethyl ethanoate). Close the chamber and wait until the solvent front has risen to within a few millimetres of the top of the plate.
20. Remove the plate from the chamber, immediately marking the position of the solvent front, and allow it to dry.
21. Place the TLC plate in a beaker containing a few iodine crystals and cover the beaker with a clock glass. Once any brownish spots appear, remove the plate and lightly mark the observed spots with a pencil. Alternatively, observe the dried TLC plate under UV light and lightly mark with a pencil any spots observed.
22. Calculate the R_f values of the spots. This will give you some indication of the purity of the aspirin you have prepared.

AH Chem Practicals

7B

Determination of Aspirin

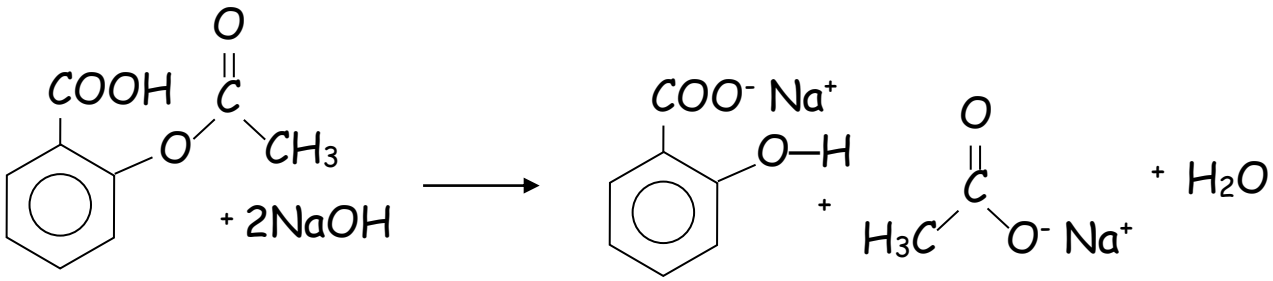
Introduction



Aspirin has the following structural formula:

Since it is insoluble in water, aspirin has to be determined by a back titration technique. This involves treating a sample of accurately known mass with a definite amount of sodium hydroxide, i.e. the volume and concentration of the alkali must be accurately known. The alkali first catalyses the hydrolysis of the aspirin to ethanoic and salicylic acids and then neutralises these acids.

The overall equation for the reaction is:



An excess of alkali has to be used and the amount remaining after reaction is determined by titrating it against a standard solution of sulfuric acid. It is good practice, especially when using an unfamiliar procedure, to carry out a control experiment. In this case the control would involve carrying out the determination of aspirin using a pure sample of the compound. If the mass of aspirin you determine matches the mass you started with then this establishes the validity of the procedure and the results. However, if the experimental result deviates significantly from the true value then this could arise from bad technique or not using standardised solutions. These factors should be checked before dismissing a procedure as invalid.

Requirements

250cm^3 standard flasks
conical flasks (100cm^3 and 250cm^3)
25cm^3 pipette
50cm^3 burette
weighing bottle
balance (accurate to 0.01g)
hot plate (or Bunsen burner and tripod)
50cm^3 measuring cylinder
100cm^3 beakers
pipette filler

aspirin tablets
sample of pure aspirin
standardised 0.050mol l^{-1} sulfuric acid
standardised 1.0mol l^{-1} sodium hydroxide
phenolphthalein
deionised water
filter funnel
white tile
wash bottle
dropper

Hazcon

Wear eye protection and if any chemical splashes on the skin, wash it off immediately.

0.50mol l^{-1} sulfuric acid irritates the eyes and skin.

1.0mol l^{-1} sodium hydroxide is corrosive to the eyes and skin. Gloves and goggles should be worn.

Phenolphthalein indicator solution is highly flammable and irritating to the eyes because of its ethanol content.

Aspirin irritates the eyes and skin.

Procedure

Control experiment using pure aspirin

1. Add about 1.5g of pure aspirin to the weighing bottle and weigh the bottle and contents.
2. Transfer the pure aspirin to a large conical flask and reweigh the weighing bottle.
3. Rinse the 25cm^3 pipette with 1.0mol l^{-1} sodium hydroxide and pipette 25cm^3 of this solution into the flask containing the pure aspirin.
4. To the mixture in the flask, add approximately 25cm^3 of deionised water.
5. Place the flask on the hot plate and simmer the mixture very gently for about 30 minutes.
6. Allow the reaction mixture to cool before transferring it to the 250cm^3 standard flask.
7. Rinse the conical flask with a little deionised water and add the rinsings to the standard flask. Repeat this procedure several times and add the rinsings to the flask. Make up the solution to the graduation mark with deionised water.
8. Stopper the flask and invert it several times to ensure the contents are completely mixed.
9. Rinse the burette, including the tip, with 0.050mol l^{-1} sulfuric acid and fill it with the same solution.
10. Rinse the 25cm^3 pipette with the 'standard flask' solution and pipette 25cm^3 of it into a 100cm^3 conical flask.
11. Add a few drops of phenolphthalein indicator to the solution and titrate to the end-point.
12. Repeat the titrations until two concordant results are obtained.
13. Calculate the mass of aspirin in the initial sample using the accurate concentrations of the sulfuric acid and sodium hydroxide solutions provided by your practitioner.
14. Compare your result with the initial mass of pure aspirin you used.

Determination of aspirin in a commercial tablet

1. Add a definite number of aspirin tablets (about 1.5g in mass) to the weighing bottle and weigh the bottle and contents.
2. Transfer the tablets to a large conical flask and reweigh the weighing bottle.
3. Repeat steps 3 to 12 of the above procedure.
4. Calculate the mass of aspirin per tablet using the accurate concentrations of the sulfuric acid and sodium hydroxide solutions provided by your practitioner.
5. Compare your result with the manufacturer's specification.

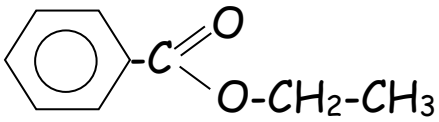
AH Chem Practicals

8

Hydrolysis of Ethyl Benzoate

Introduction

Benzoic acid can be prepared by the alkaline hydrolysis of the ester, ethyl benzoate:



If sodium hydroxide is used, then the residual solution will contain sodium benzoate. Insoluble benzoic acid can be displaced from this solution by acidification. It can then be filtered off and purified by recrystallisation. The percentage yield of benzoic acid can be calculated. The purity and identity of the final sample can be checked by measuring its melting point and mixed melting point, and by thin-layer chromatography.

Requirements

ethyl benzoate
2mol l^{-1} sodium hydroxide
5mol l^{-1} hydrochloric acid
blue litmus paper or pH paper
anti-bumping granules
deionised water
sample of pure benzoic acid
iodine
dichloromethane
ethyl ethanoate
Buchner funnel and flask
water pump
filter papers
clock glass
glass stirring rod
dropper
oven
100cm^3 round-bottomed flask
cork ring
condenser
heating mantle
100cm^3 measuring cylinder
250cm^3 beaker
glass filter funnel
thermometer
balance (accurate to 0.01g)
hot plate
capillary tubes
melting point apparatus
chromatography chamber
TLC plate
test-tubes
UV lamp

Hazcon

Wear eye protection and if any chemical splashes on the skin, wash it off immediately.
Ethyl benzoate is of low volatility and flammability. It irritates the eyes and is harmful if ingested in quantity.
2 mol l^{-1} sodium hydroxide is corrosive to the eyes and skin. Gloves and goggles should be worn.
5 mol l^{-1} hydrochloric acid is irritating to the eyes, lungs and skin. Wear gloves.
Benzoic acid is of low volatility and flammability. It may be harmful if ingested in quantity.
Dichloromethane irritates the eyes and skin, and is at its most harmful if inhaled. Wear gloves.
Ethyl ethanoate is irritating to the eyes, is volatile and can irritate the respiratory system. It is highly flammable. Wear gloves.

Procedure

1. Weigh a 100cm^3 round-bottomed flask supported on a cork ring. To the flask add about 5g of ethyl benzoate and reweigh the flask and its contents.
2. To the ethyl benzoate add approximately 50cm^3 of 2mol l^{-1} sodium hydroxide and a few anti-bumping granules.
3. Set up the apparatus for heating under reflux. Using a heating mantle, reflux the reaction mixture until all the oily drops of the ester have disappeared. This may take 45–60 minutes.
4. Allow the apparatus to cool and then transfer the reaction mixture to a 250cm^3 glass beaker.
5. Slowly and with stirring add 5 mol l^{-1} hydrochloric acid to the reaction mixture to precipitate out the benzoic acid. Continue adding the acid until no more precipitation takes place and the mixture turns acidic (test with blue litmus paper or pH paper). About 30cm^3 of acid will be required.
6. Allow the mixture to cool to room temperature and filter off the precipitate at the water pump. Wash the crude benzoic acid with a small volume of water.
7. Transfer the crude benzoic acid to a 250cm^3 beaker and recrystallise it from about 100cm^3 of water.
8. Filter off the crystals of benzoic acid at the water pump and wash them with a small volume of water. Allow air to be drawn through the crystals for a few minutes in order to partially dry them.
9. Weigh a clock glass and transfer the crystals to it. Dry the crystals in an oven at about 70°C and then reweigh the clock glass and crystals.
10. Calculate the percentage yield of benzoic acid.
11. Determine the melting point of the benzoic acid product.
12. Grind a 50:50 mixture of your product and a pure sample of benzoic acid, and determine the mixed melting point. This will give you some indication of the purity of the benzoic acid you prepared.
13. Take a TLC plate and using a pencil lightly draw a line across the plate about 1cm from the bottom. Mark two well-spaced points on the line.
14. Place small amounts (about a third of a spatulaful) of your benzoic acid product and a pure sample of benzoic acid in two separate test-tubes.
15. Add about 1cm^3 of ethyl ethanoate to each of the test-tubes to dissolve the benzoic acid samples.
16. Use capillary tubes to spot each of the two samples onto the TLC plate. Allow to dry and repeat two or three more times.
17. After the spots have dried, place the TLC plate into the chromatography chamber, making sure that the pencil line is above the level of the solvent (dichloromethane). Close the chamber and wait until the solvent front has risen to within a few millimetres of the top of the plate.
18. Remove the plate from the chamber, immediately marking the position of the solvent front, and allow it to dry.
19. Place the TLC plate in a beaker containing a few iodine crystals and cover the beaker with a clock glass. Once any brownish spots appear, remove the plate and lightly mark with a pencil the observed spots. Alternatively, observe the dried TLC plate under UV light and lightly mark with a pencil any spots observed.
20. Calculate the R_f values of the spots. This will give you some indication of the purity of the benzoic acid you have prepared.

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9

Preparation of Ethyl Ethanoate

Introduction

The ester ethyl ethanoate can be prepared by the condensation reaction between ethanoic acid and ethanol in the presence of concentrated sulphuric acid. The latter catalyses the reaction by supplying protons. The product can be separated from the reaction mixture by distillation and after purification it can be weighed and the percentage yield determined.

Requirements

$100cm^3$ round-bottomed flask
$50cm^3$ round-bottomed flasks
cork ring
condenser
still head
receiver adapter
thermometer adapter
thermometer
balance (accurate to 0.01 g)
heating mantle
ethanol
glacial ethanoic acid
concentrated sulfuric acid
2 mol l_{-1} sodium carbonate
calcium chloride solution (10g in $10cm^3$ water)
anhydrous calcium chloride
anti-bumping granules
$100cm^3$ separating funnel
$10cm^3$ measuring cylinder
$50cm^3$ conical flasks

Hazcon

Wear eye protection and if any chemical splashes on the skin, wash it off immediately.
Concentrated sulfuric acid causes severe burns to the eyes and skin. Wear goggles and gloves.
Liquid ethanoic acid and its vapour cause severe burns to the eyes and skin. Wear goggles and gloves.
Ethanol is volatile and highly flammable, is irritating to the eyes and intoxicating if inhaled or ingested.
2mol l^{-1} sodium carbonate is irritating to the eyes.
Anhydrous calcium chloride irritates the eyes, lungs and skin. Wear gloves.
The product, ethyl ethanoate, is highly flammable and irritates the eyes and respiratory system.

Procedure

1. Weigh a 100cm^3 round-bottomed flask supported on a cork ring. To the flask add approximately 20cm^3 of ethanol and reweigh the flask and its contents.
2. To the ethanol add about 20cm^3 of ethanoic acid.
3. Carefully add approximately 4cm^3 of concentrated sulfuric acid and swirl the contents of the flask.
4. Add a few anti-bumping granules to the reaction mixture and set up the apparatus for heating under reflux. Gently reflux the mixture for about 10 minutes.
5. Allow the apparatus to cool slightly and then rearrange it for distillation and distil off about two-thirds of the mixture.
6. Pour the distillate into a separating funnel and add about 10cm^3 of 2mol l^{-1} sodium carbonate. Stopper the funnel and carefully shake the contents, opening the tap at frequent intervals to release the pressure of the evolved carbon dioxide. This process removes acidic impurities.
7. Clamp the separating funnel and allow the two layers to separate.
8. Remove the stopper from the funnel and run off the lower aqueous layer into a beaker and dispose of it down the sink. To the remaining organic layer add the calcium chloride solution and shake vigorously. This removes any remaining ethanol.
9. After allowing the mixture to separate, run off and discard the lower aqueous layer. Transfer the organic layer (the crude ethyl ethanoate) into a small conical flask and add a few pieces of anhydrous calcium chloride. Stopper the flask and shake the mixture for a few minutes until the liquid is clear.
10. Weigh a dry 50cm^3 round-bottomed flask in which to collect the pure ethyl ethanoate.
11. Decant the ethyl ethanoate into another dry 50cm^3 round-bottomed flask and add a few anti-bumping granules. Distil the ethyl ethanoate very slowly, collecting the liquid which comes over between 74°C and 79°C in the pre-weighed flask. To cut down loss of the volatile ethyl ethanoate during distillation, the receiving flask could be placed in an ice bath.
12. Weigh the flask and product.
13. Calculate the percentage yield.

Colorimetric Determination of Mn

Introduction

Colorimetry is an analytical technique used to determine the concentrations of coloured substances in solution. It relies on the fact that a coloured substance absorbs light of a colour complementary to its own and the amount of light it absorbs (absorbance) is proportional to its concentration.

Colorimetry is particularly suited to the determination of manganese in steel because the manganese can be converted into permanganate ions, which are coloured. The conversion is achieved in two stages. Using nitric acid, the managanese is first oxidised to manganese(II) ions, which are then oxidised to permanganate ions by the more powerful oxidising agent, potassium periodate.

Requirements

acidified potassium periodate	5 g potassium periodate per 100 cm^3 of 2 mol l^{-1} nitric acid	$50cm^3$ burette
standardised 0.0010 mol l^{-1} acidified potassium permanganate		standard flasks ($50cm^3$ and $100cm^3$)
propanone		balance (accurate to 0.001g)
2mol l^{-1} nitric acid		glass beakers ($50cm^3$ and $250cm^3$)
85% phosphoric acid		Bunsen burner, heating mat and tripod
potassium persulfate		measuring cylinders ($50cm^3$ and $10cm^3$)
steel paper clips		clock glass
deionised water		filter funnel
anti-bumping granules		tweezers
colorimeter		wash bottle
green filter		dropper
optically matched cuvettes		wire cutters

Hazcon

Wear eye protection and if any chemical splashes on your skin wash it off immediately.

The acidified 0.0010mol l^{-1} potassium permanganate is harmful if ingested and irritates the eyes and skin. Wear gloves.

Both 2mol l^{-1} nitric acid and its vapour are corrosive and toxic, causing severe burns to the eyes, digestive and respiratory systems. Wear gloves.

85% phosphoric acid is corrosive: it burns and irritates the eyes and skin. It is a systemic irritant if inhaled and if swallowed causes serious internal injury. Wear gloves.

Acidified potassium periodate solution is harmful if swallowed and is an irritant to the eyes, skin and respiratory system. It is also corrosive. Wear gloves.

Potassium persulfate is harmful if swallowed or inhaled as a dust. It irritates the eyes, skin and respiratory system, causing dermatitis and possible allergic reactions. Wear gloves.

Propanone is volatile and highly flammable, and is harmful if swallowed. The vapour irritates the eyes, skin and lungs, and is narcotic in high concentrations. Wear gloves.

Procedure

Part A – Calibration graph

1. Rinse the burette, including the tip, with 0.0010mol l^{-1} acidified potassium permanganate and fill it with the same solution.
2. Run 2cm^3 of the permanganate solution into a 50cm^3 standard flask and make up to the graduation mark with deionised water.
3. Stopper the flask and invert it several times to ensure the contents are completely mixed.
4. Rinse a cuvette with some of the solution and fill it.
5. Using a colorimeter (fitted with a green filter) measure the absorbance of the solution in the cuvette. If you have more than one green filter, choose the one that gives maximum absorbance.
6. Repeat steps 2 to 5 with 4, 6, 8, 10, 12 and 14 cm_3 of the permanganate stock solution in the burette.
7. Plot a calibration graph of 'absorbance' against 'concentration of potassium permanganate'. Your practitioner will provide you with the accurate concentration of the acidified potassium permanganate stock solution.

Part B – Conversion of manganese to permanganate

1. Degrease a steel paper clip by swirling it with a little propanone in a beaker. Using tweezers remove the paper clip and leave it to dry for a minute or so on a paper towel.
2. Cut the paper clip into small pieces.
3. Weigh **accurately** about 0.2g of the paper clip pieces and transfer them to a 250cm^3 glass beaker.
4. Add approximately 40cm^3 of 2mol l^{-1}nitric acid to the beaker and cover it with a clock glass.
5. Heat the mixture cautiously, in a fume cupboard, until the reaction starts. Continue heating gently to maintain the reaction, but remove the source of heat if the reaction becomes too vigorous.
6. Once the steel has reacted, allow the solution to cool a little. Add a couple of anti-bumping granules and then boil the solution until no more brown fumes are given off.
7. Once this solution has cooled considerably – no more than 'hand hot' – add about 5cm^3 of 85% phosphoric acid, approximately 0.2g of potassium persulfate and a couple of fresh anti-bumping granules. Boil the mixture for about 5 minutes.
8. To this solution, add approximately 15cm^3 of acidified potassium periodate solution plus a couple of fresh anti-bumping granules and then gently boil the mixture. The solution will start to turn pink. Continue gently boiling until the intensity of the pink colour remains constant. This should take about 5 minutes.
9. Allow the pink solution to cool to room temperature and then transfer it to a 100cm^3 standard flask, leaving the anti-bumping granules in the beaker.
10. Rinse the beaker several times with a little deionised water and add the rinsings (but not the anti-bumping granules) to the flask.
11. Make up the solution to the graduation mark with deionised water.
12. Stopper the flask and invert it several times to ensure the contents are completely mixed.
13. Using a colorimeter fitted with the appropriate green filter, measure the absorbance of the solution.
14. Use your calibration graph to convert the absorbance to a permanganate concentration and then calculate the percentage by mass of manganese in the steel paper clip.

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11

Preparation of Cyclohexene from Cyclohexanol

Introduction

Cyclohexene can be prepared by dehydrating cyclohexanol using concentrated phosporic acid. The product can be separated from the reaction mixture by distillation, and after purification it can be weighed and the percentage yield determined.

Requirements

cyclohexanol
85% phosphoric acid
saturated sodium chloride solution
anhydrous calcium chloride
anti-bumping granules
bromine solution
250 cm^3 separating funnel
10 cm^3 measuring cylinder
50 cm^3 conical flask
dropper
test-tube and rack
50 cm^3 round-bottomed flasks
condenser
still head

50 cm^3 round-bottomed flasks
cork ring
condenser
still head
receiver adapter
thermometer adapter
thermometer
balance (accurate to 0.01 g)
heating mantle
receiver adapter anti-bumping granules
thermometer adapter bromine solution
thermometer
balance (accurate to 0.01 g)
heating mantle

Hazcon

Wear eye protection and if any chemical splashes on the skin, wash it off immediately.
Cyclohexanol (including its vapour) is harmful to the eyes, lungs and skin, and is harmful if swallowed. It is flammable and is a suspected carcinogen. Wear gloves.
85% phosphoric acid is corrosive; it burns and irritates the eyes and skin. It is a systemic irritant if inhaled and if swallowed causes serious internal injury. Wear gloves.
Anhydrous calcium chloride irritates the eyes, lungs and skin. Wear gloves.
The product, cyclohexene, is highly flammable and its vapour is moderately toxic to the eyes, skin and respiratory system. Wear gloves. At the end of the experiment, dispose of the cyclohexene since it may form unstable peroxides if stored.
Bromine solution causes burns and is toxic. Wear gloves.

Procedure

1. Weigh a 50cm^3 round-bottomed flask supported on a cork ring. To the flask add approximately 20g of cyclohexanol and reweigh the flask and its contents.
2. To the cyclohexanol add dropwise with swirling about 8cm^3 of 85% phosphoric acid.
3. Add a few anti-bumping granules to the reaction mixture and set up the apparatus for distillation. Gently heat the mixture for about 15 minutes making sure it doesn't boil. Raise the temperature and distil the mixture very slowly, collecting the liquid which comes over between 70°C and 90°C.
4. Pour the distillate into a separating funnel and add about an equal volume of saturated sodium chloride solution. Stopper the funnel and shake the contents vigorously.
5. Clamp the separating funnel and allow the two layers to separate.
6. Remove the stopper from the funnel and run off the lower aqueous layer into a beaker and dispose of it down the sink.
7. Run the top layer (the crude alkene) into a small conical flask and add a few pieces of anhydrous calcium chloride. Stopper the flask and shake the mixture for a few minutes until the liquid is clear.
8. Weigh a dry 50cm^3 round-bottomed flask in which to collect the pure cyclohexene.
9. Decant the alkene into another dry 50cm^3 round-bottomed flask and add a few anti-bumping granules. Distil the alkene very slowly, collecting the liquid which comes over between 81°C and 85°C in the preweighed flask. To cut down loss of the volatile cyclohexene during distillation, the receiving flask could be placed in an ice bath.
10. Weigh the flask and product.
11. Carry out a test to show that the product is unsaturated.
12. Calculate the percentage yield.

1	