Durham Johnston Comprehensive School Chemistry Department

Organic Synthesis Practical Record Book

A-Level Chemistry

I Gibb - Science

Class:

Contents

Common Practical Assessment Criteria (CPAC)

Students have to meet the expectations of the Common Practical Assessment Criteria (CPAC). Students are expected to develop these competencies through the acquisition of technical skills demonstrated in practical activity undertaken during the course of study,

Teachers who award a pass need to be confident that the student consistently and routinely exhibits the competencies listed below before completion of the A-Level course.

CPAC Statements:

(1) Follows written procedures	Correctly follows instructions to carry out the experimental techniques or procedures.
(2) Applies investigative approaches and methods when using instruments and equipment	Correctly uses appropriate instrumentation, apparatus and materials (including ICT) to carry out investigative activities, experimental techniques and procedures with minimal assistance or prompting. Carries out techniques or procedures methodically, in sequence and in combination, identifying practical issues and making adjustments when necessary. Identifies and controls significant quantitative variables where applicable, and plans approaches to take account of variables that cannot readily be controlled. Selects appropriate equipment and measurement strategies in order to ensure suitably accurate results.
(3) Safely uses a range of practical equipment and materials	Identifies hazards and assesses risks associated with these hazards when carrying out experimental techniques and procedures in the lab or field. Uses appropriate safety equipment and approaches to minimise risks with minimal prompting. Identifies safety issues and makes adjustments when necessary.
(4) Makes and records observations	Makes accurate observations relevant to the experimental or investigative procedure. Obtains accurate, precise and sufficient data for experimental and investigative procedures and records this methodically using appropriate units and conventions.
(5) Researches, references and reports	Uses appropriate software and/or tools to process data, carry out research and report findings. Sources of information are cited demonstrating that research has taken place, supporting planning and conclusions.

Risk Assessment

What is risk assessment?

A risk assessment is a judgment of how likely it is that someone (anyone) might come to harm if a planned action is carried out. The law requires the likelihood of harm to be reduced to as low as is reasonably practicable. Risk assessments, although an excellent idea for all of us, are only *legally* required for actions which take place at work. The *significant findings* of risk assessment must be recorded. (You must show the answer, you don't have to show your workings).
You carry out risk assessments all the time, for example, when riding a bike or crossing the road. When riding a bike in the UK you can choose whether or not to wear a cycling helmet. However, because risk assessments are required at work, paid cycling couriers will wear helmets. The risk of them being knocked off their bike is quite high but a helmet reduces the likelihood of head injury.

How do you 'do' a risk assessment?

To make a risk assessment you need to know the **hazards** and the **risk** of them causing harm in the planned activity.
A **hazard** is anything which could cause harm. For example some chemicals, electricity at high enough currents, glass (if it breaks) and even you running in the corridor are all hazards because they can all cause harm. Although sometimes you can use your common sense to identify a hazard, often you will need specialist information, eg as provided on *CLEAPSS Student Safety Sheets* or on chemical suppliers' *Safety Data Sheets*.
The **risk** is the likelihood that a hazard would cause significant harm. It is a matter of judgment and depends on:
• how *likely* it is that something would go wrong with this hazard;
• how *serious* any resulting injuries would be; and
• how *many* people would be affected.
To reduce the risks to an acceptable level, we put in place relevant **control measures**. These are the safety precautions used to reduce the risk of harm. In science we often wear safety spectacles, or use fume cupboards. We also minimise the quantities of materials used and the concentration of hazardous solutions.

What should you do when making a risk assessment?

When making a risk assessment, go through the following process.
1. Consider what materials you are working with and what procedures you are you following. You could list them on the CLEAPSS *Student Form for Assessing Risk*. Think about microorganisms, heavy weights, electricity, chemicals (how much of each, what concentration of solutions), hot objects. You should also try to find out if there are any hazardous materials produced by your procedure - you may need to ask your teacher!
2. For each of the materials and procedures, ask what are the hazards? Add them to your list. What could possibly go wrong? Look up the materials and procedures in reliable and relevant sources, eg, *CLEAPSS Student Safety Sheets*.
3. How many people could be affected if it went wrong? Who would they be?
4. What control measures (safety precautions) would you adopt? Check relevant *CLEAPSS Student Safety Sheets*.
5. Make sure you record anything important and especially the control measures.
6. Have the result of the risk assessment checked by your teacher before you carry on.

Sample Risk Assessment Table

Proposed Experiment/Activity:

Hazardous chemical being used or made, or hazardous procedure or equipment.	Nature of the hazard (s)	Source(s) of information	Control measures to reduce risk.

Checked by: Date:

Key Deadlines

Task	Date Due
Experiment 1	
Experiment 2	
Experiment 3	
Experiment 4	
Experiment 5	
Pharmaceutical Poster Task	

Practical Preparation Tasks

Prior to each experiment, one or more preparation exercises must be completed. These may involve reading, watching video clips, writing a risk assessment, answering questions or using interactive software to rehearse techniques. The instructions for these exercises will be in this booklet and on the Student Home Area (S: drive) [S: → Science → Students → Chemistry → Organic Synthesis Summer 2019]

Preparation tasks are a key part in your demonstration of competence of CPAC tasks, particularly tasks 2 and 5. They must be completed prior to beginning the practical task they relate to.

Recording your work

For each experiment, you must keep a record of your work, and these must be handed in with your lab book. Each page of your records should be titled with the experiment title. The names of lab partners or group members should also be given, and your work should be dated on every page. If a second page is required, begin it with the experiment title and date.

Observations, measurements and data should be recorded immediately and in full (with units, where relevant. Your records should show exactly what was measured and recorded, not what was predicted! When working in pairs or groups, individual records should be kept at the time of the experiment.

Some experiments require an experiment plan to be written prior to the lab session, these should be part of your records and handed in as such.

Experiment 1 - Virtual Synthesis of Aspirin

In this first task, you will use the Royal Society of Chemistry's Lab Primer to work through the synthesis of Aspirin.

The Royal Society of Chemistry's aspirin screen experiment is a freely available digital resource. It is designed to enhance students understanding of organic chemistry and improve practical skills. The interactive screen experiments enable students to undertake an aspirin synthesis, perform recrystallization, thin layer chromatography and modify experimental conditions to determine the effect on yield.

You will need to go to the website below:

http://www.rsc.org/learn-chemistry/resources/screen-experiment/aspirin/experiment/1

and register to create an account. The primer consists of 4 sections for you to work through. You will need to download and print your virtual lab book, along with your scores and badges, then hand this in to demonstrate you have completed this exercise.

This task must be completed and handed in by …….

Student Records

Experiment 2 - Extracting limonene from oranges by steam distillation

This experiment demonstrates the extraction of plant oils.

The peel of **oranges** is boiled in water and the oil produced (**limonene**) distilled in steam at a temperature just below 100 °C, well below its normal boiling point. The immiscible oil can then be separated. Direct extraction by heating would result in decomposition whereas **steam distillation** does not destroy the chemicals involved.

The experiment also links for tests for **unsaturation.**

Preparation Tasks:

Before beginning this task, ensure that you have visited the interactive primer and researched:

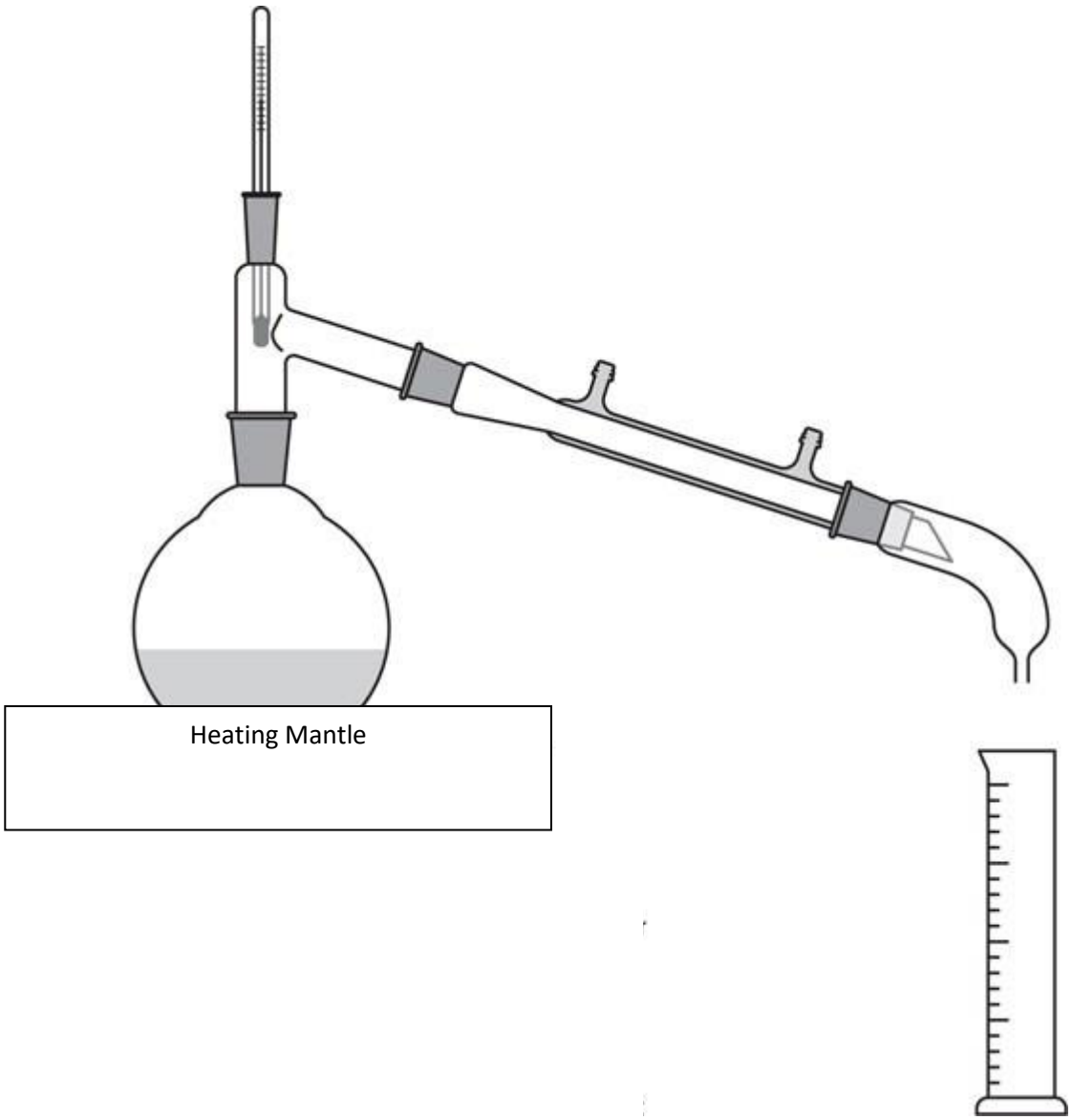
Steam Distillation http://www.rsc.org/learn-chemistry/resource/res00002249/steam-distillation and Distillation http://www.rsc.org/learn-chemistry/resource/res00001070/distillation#!cmpid=CMP00001936.

You must also complete a risk assessment for your experiment, in the space provided.

Apparatus and chemicals

- Eye protection
- Grater
- Heating Mantle
- Clamps, stands and bosses
- Oranges, 2
- 110 °C thermometer
- Measuring cylinder (100 cm^3)
- Measuring cylinder (50 cm^3)
- Distillation apparatus
- 250 cm^3 round bottomed flask
- Still head
- Thermometer pocket
- Condenser
- Receiver adapter
- Test tubes and bungs, 3
- Dropping pipette
- Anti-bumping granules
- Bromine water, no more than 0.2% v/v (**Irritant**)
- Distilled water, 100 cm^3.

Apparatus



Risk Assessment

Procedure

a Grate the outer orange coloured rind of two oranges and add to 100 cm^3 of distilled water in the 250 cm^3 round bottomed flask. Add anti-bumping granules to the round bottomed flask.

b Set up the distillation apparatus as shown in the apparatus section.

c Heat the flask so that distillation proceeds at a steady rate, approximately one drop per second of distillate. (Note: Take care not to let the liquid in the round bottomed flask boil too strongly).

d Collect approximately 50 cm^3 of distillate in the measuring cylinder. The oil layer will be on the surface.

e Using a dropping pipette carefully remove the oil layer into a test tube for the next stage.

f Cautiously smell the extracted oil by wafting the fumes towards the nose. Do not breathe in directly from the test tube.

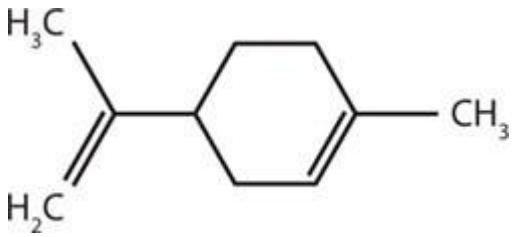
Action of bromine water

g Measure out approximately 1 cm^3 of bromine water into each of three test tubes.

h Add a few drops of the limonene oil to one test tube, a few drops of cyclohexane to another, and a few drops of cyclohexene to the third. Place in the bungs and agitate. If the bromine water is decolourised the molecule contains double bonds.

Additional Information

Limonene (1-methyl-4-prop-1-en-2-yl-cyclohexene) is an unsaturated hydrocarbon, classed as a terpene. At room temperature it is a colourless oily liquid with the smell of oranges. Its molecular formula is $C_{10}H_{16}$ and its boiling point is 176 °C.



Limonene is a chiral molecule with two optical isomers (enantiomers). The major biological form *d*limonene, the (*R*)-enantiomer, is used in food manufacture and medicines. It is also used as a fragrance in cleaning products, a botanical insecticide, and due to its flammability, a potential biofuel.

The (*S*)-enantiomer, *l*-limonene, is also used as a fragrance but has a piney, turpentine odour.

Student Records

Extraction of Limonene – Student Feedback Sheet

Practical skills and techniques covered in this task:		**Awarded**
Use and application of scientific methods and practices		
1.2.1 (b)	safely and correctly use a range of practical equipment and materials, including identification of potential hazards. Learners should understand how to minimise the risks involved.	
1.2.1 (c)	follow written instructions	
1.2.1 (d)	make and record observations/measurements.	
1.2.1 (e)	keep appropriate records of experimental activities	
1.2.1 (f)	present information and data in a scientific way	
1.2.1 (h)	use online and offline research skills including websites, textbooks and other printed scientific sources of information	
1.2.1 (i)	correctly cite sources of information	
Practical Techniques		
1.2.2 (a)	use of appropriate apparatus to record a range of measurements (to include mass, time, volume of liquids and gases, temperature)	
1.2.2 (b)	use of a water bath or electric heater or sand bath for heating	
1.2.2 (d)	use of laboratory apparatus for a variety of experimental techniques including: **(ii)** distillation and heating under reflux, including setting up glassware using retort stand and clamps **(iii)** qualitative tests for ions and organic functional groups	

Further comment and guidance, including recovery tasks.

Experiment 3 - Coursework Conundrum

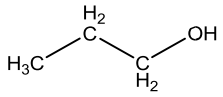
Preparatory Task 1

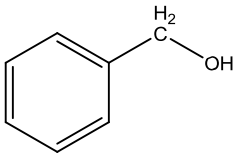
(Remember to give full references for any information beyond A-level that you find out)

Acidified potassium dichromate is a common oxidising agent for alcohols. In the process of the reaction $Cr_2O_7^{2-}$ is reduced to Cr^{3+}. Write two half equations for the oxidation of ethanol to ethanoic acid using acidified potassium dichromate [HINT: In the oxidation process, the carbon bonded to the oxygen is the atom undergoing oxidation (oxidation state –1 to +3)].

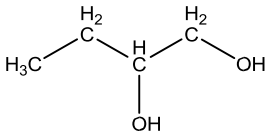
Combine the two half equations to give the full redox equation for the reaction.

1. For each of the alcohols below, name the alcohol and draw and name the product obtained from the oxidation of the alcohol after refluxing in excess acidified potassium dichromate.

a) 

b) 

c) $CH_3CH(OH)CH_3$

d) 

2. Impure solids can be purified by recrystallisation. Outline the steps involved in the purification of a solid by recrystallisation

Generally, organic compounds are insoluble in polar solvents such as water and ethanol. However carboxylic acids are an exception, with short chain carboxylic acids being totally miscible with both water and ethanol. Use your understanding of the interactions involved to explain the data below (taken from http://www.auburn.edu/~deruija/pda1_acids1.pdf)

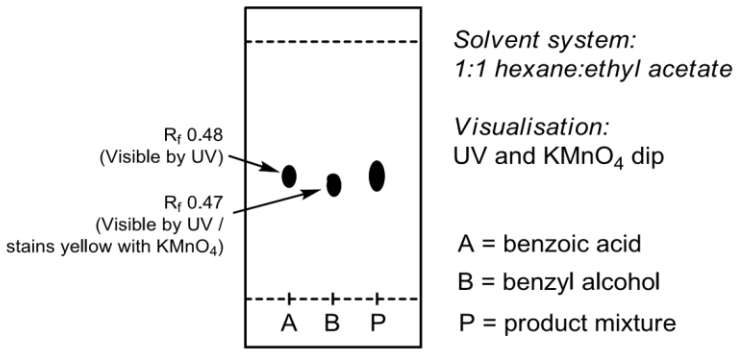
RCOOH; R=	Solubility in water / g per 100 cm^3	Solubility in ethanol / g per 100 cm^3
H-	soluble	soluble
CH_3-	soluble	soluble
CH_3CH_2-	soluble	soluble
$CH_3CH_2CH_2$-	soluble	soluble
$CH_3CH_2CH_2CH_2$-	3.7	soluble
$CH_3CH_2CH_2CH_2CH_2$-	1.0	soluble
C_6H_5-	0.34	soluble
$CH_3(CH_2)_8$-	0.015	soluble
$CH_3(CH_2)_{10}$-	Insoluble	100
$CH_3(CH_2)_{12}$-	Insoluble	soluble
$CH_3(CH_2)_{16}$-	Insoluble	5.0

72 Elms Road
Hayle
HE2 3GF

Dear scientist,

Help! My A-level chemistry coursework is due in at the end of the week and I have a problem. My product is not pure ;-(

The coursework practical involved the oxidation of benzyl alcohol to form benzoic acid by refluxing the alcohol in acidified potassium dichromate overnight. However, when isolated my reaction has not gone to completion and my product is not clean. When I run a TLC of my product, despite appearing to be only one spot by TLC analysis, staining with $KMnO_4$ reveals that it is in fact a mixture. My product is also slightly green indicating the presence of left over Cr^{3+} salts plus I think I left some of the anti-bumping granules in the mixture as well!



How can I purify my product? I have included a sample of my product mixture with this letter. I know this is cheating but can you check that my product is indeed impure by repeating the TLC and then purify it for me please. As I will need to submit details of my practical please also provide a full procedure and rationale for the method used together with a sample of pure benzoic acid and a TLC plate showing its purity.

You're a lifesaver ;-)

L. A. Zyudent

L. A. Zyudent

Preparatory Task 2

You will need to write a practical procedure for this experiment, and a risk assessment.

Please ensure that you have visited the lab primer and researched TLC http://www.rsc.org/learn-chemistry/resource/res00001074/thin-layer-chromatography and recrystallization http://www.rsc.org/learn-chemistry/resource/res00001065/recrystallisation

Risk Assessment:

Student Notes:

Please glue in a photo of your TLC plate prior to purification here.

Please glue in a photo of your TLC plate after purifying here.

Student Records

Coursework Conundrum – Student Feedback Sheet

Practical skills and techniques covered in this task:		**Awarded**
Independent thinking		
1.2.1 (a)	apply investigative approaches and methods to practical work	
Use and application of scientific methods and practices		
1.2.1 (b)	safely and correctly use a range of practical equipment and materials, including identification of potential hazards. Learners should understand how to minimise the risks involved.	
1.2.1 (d)	make and record observations/measurements.	
1.2.1 (e)	keep appropriate records of experimental activities	
1.2.1 (f)	present information and data in a scientific way	
Research and referencing		
1.2.1 (h)	use online and offline research skills including websites, textbooks and other printed scientific sources of information	
1.2.1 (i)	correctly cite sources of information	
Practical Techniques		
1.2.2 (d)	use of laboratory apparatus for a variety of experimental techniques including: **(iv)** filtration, including use of fluted filter paper, or filtration under reduced pressure	
1.2.2 (g)	purification of: **(i)** a solid product by recrystallisation	
1.2.2 (i)	use of thin layer chromatography	

Further comment and guidance, including recovery tasks.

Experiment 4 - Preparation of Benzoic Acid

Preparatory Tasks

Before beginning the procedure, you must complete a risk assessment.

You should make sure you have visited the Lab Primer and researched heating under Refulx http://www.rsc.org/learn-chemistry/resource/res00001075/heating-under-reflux and filtering under reduced pressure http://www.rsc.org/learn-chemistry/resource/res00001072/vacuum-filtration#!cmpid=CMP00001938

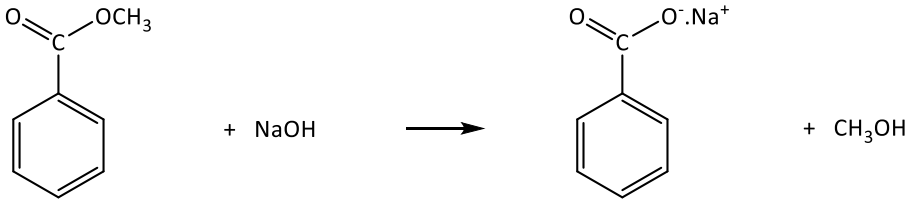
Chemical List:

Label	Identity	Hazard information
methyl benzoate	methyl benzoate, $C_6H_5COOCH_3(l)$	*Use CLEAPSS resources or similar to find this information*
2.0 mol dm^{-3} NaOH(aq)	2.0 mol dm^{-3} aqueous sodium hydroxide, NaOH(aq)	*Use CLEAPSS resources or similar to find this information*
2.00 mol dm^{-3} HCl(aq)	2.00 mol dm^{-3} aqueous hydrochloric acid, HCl(aq)	*Use CLEAPSS resources or similar to find this information*
ethanol	ethanol, $C_2H_5OH(l)$	*Use CLEAPSS resources or similar to find this information*
methyl orange indicator	0.1% methyl orange indicator	*Use CLEAPSS resources or similar to find this information*

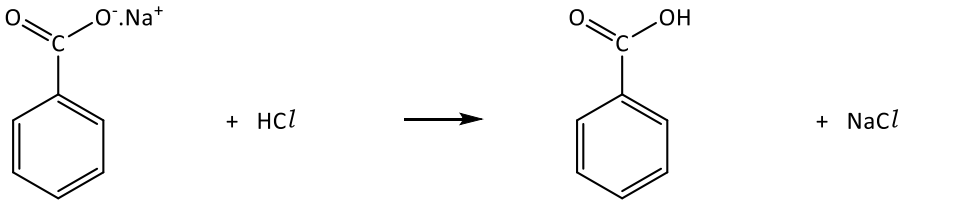
Introduction

You will prepare benzoic acid, C_6H_5COOH, by alkaline hydrolysis of an ester, followed by acidification.

1. Alkaline hydrolysis of an ester, methyl benzoate ($C_6H_5COOCH_3$), to form a solution of sodium benzoate (C_6H_5COONa), a salt of benzoic acid.
2. Acidification of sodium benzoate to form benzoic acid.



methyl benzoate + sodium hydroxide → sodium benzoate + methanol



sodium benzoate + hydrochloric acid → benzoic acid + sodium chloride

Aims and *Skills*

- To carry out a two-step synthesis, purification and identification of benzoic acid
- To carry out a risk assessment
- To heat a reaction mixture under reflux
- To purify an organic solid by recrystallization

Chemicals

Label	Identity	Hazard information
methyl benzoate	methyl benzoate, $C_6H_5COOCH_3(l)$	*Use CLEAPSS resources or similar to find this information*
2.0 mol dm^{-3} NaOH(aq)	2.0 mol dm^{-3} aqueous sodium hydroxide, NaOH(aq)	*Use CLEAPSS resources or similar to find this information*
2.00 mol dm^{-3} HC*l*(aq)	2.00 mol dm^{-3} aqueous hydrochloric acid, HC*l*(aq)	*Use CLEAPSS resources or similar to find this information*
ethanol	ethanol, $C_2H_5OH(l)$	*Use CLEAPSS resources or similar to find this information*
methyl orange indicator	0.1% methyl orange indicator	*Use CLEAPSS resources or similar to find this information*

Equipment Parts 1 and 2

- eye protection
- access to balance accurate to two decimal places
- measuring cylinder (10 cm^3)
- dropping pipettes
- Quickfit apparatus:
    - pear-shaped or round-bottom flask (50 cm^3)
    - Liebig condenser and tubing
- anti-bumping granules
- wash bottle containing distilled (or de-ionised) water
- beaker (250 cm^3)
- glass rod
- apparatus for filtration under reduced pressure and appropriate filter paper
- hot plate
- sample tube and lid
- ice
- glass marker pen
- spatula
- watch glasses

Risk Assessment

Part 1: Preparation

1. Add sequentially to a round-bottom or pear-shaped flask (50 cm^3 or 100 cm^3):
    a. 2.0 cm^3 of methyl benzoate
    b. 10.0 cm^3 of 2.0 mol dm^{-3} NaOH(aq)
    c. 10.0 cm^3 of ethanol
    d. A few anti-bumping granules.
2. Fit the condenser to the flask in preparation to heat the mixture under reflux.
3. Heat the flask gently, **without allowing the contents to boil**, for about 5 minutes, followed by gentle boiling under reflux for a further 15 minutes.
4. Allow the contents of the flask to cool. Remove the condenser and decant the solution from the anti-bumping granules into a beaker.
5. Rinse the flask with distilled water, adding the rinsing solution into the beaker.
6. Add ten drops of methyl orange indicator to your solution and then acidify with 2.0 mol dm^{-3} HC*l*(aq). Solid benzoic acid, C_6H_5COOH, will crystallise.
7. Filter the mixture under reduced pressure to obtain impure benzoic acid, washing the crystals with ice cold distilled/deionized water.

Part 2: Recrystallisation and melting point

1. Transfer the impure C_6H_5COOH to a 100cm^3 beaker
2. Place the beaker onto a hot plate.
3. Heat distilled/deionized water in a separate beaker, on the hot plate.
4. Dissolve the impure benzoic acid in a minimum volume of hot distilled/deionized water:
    a. add small volumes of hot distilled/deionized water to the crystals in the beaker
    b. gently shake the beaker in the water bath to dissolve the crystals
    c. repeat until the crystals have dissolved in a **minimum volume** of hot distilled/deionized water
5. Cool the saturated solution slowly until crystals of benzoic acid form, then cool in an ice–water bath to complete the crystallisation.
   If the crystals fail to form, try scratching the inside of the tube with a glass rod, and/or stirring the solution vigorously with a glass rod for 2-3 minutes, and/or adding a small benzoic acid 'seed' crystal – discuss these methods with your teacher before attempting.
6. Filter the purified benzoic acid crystals under reduced pressure, washing with ice-cold water.
   Place the filter paper containing the benzoic acid on a watch glass and allow the product to dry in air.
7. Weigh the **dry** product and record your yield. You will need your product for TLC and melting point determination.

Analysis of results

1. Calculate the percentage yield of benzoic acid from methyl benzoate.
   Density of methyl benzoate: 1.09 g cm^{-3}

Student Records

Synthesis of Benzoic Acid – Student Feedback Sheet

Practical skills and techniques covered in this task:		**Awarded**
Use and application of scientific methods and practices		
1.2.1 (b)	safely and correctly use a range of practical equipment and materials, including identification of potential hazards. Learners should understand how to minimise the risks involved.	
1.2.1 (c)	follow written instructions	
1.2.1 (d)	make and record observations/measurements.	
1.2.1 (e)	keep appropriate records of experimental activities	
Practical Techniques		
1.2.2 (a)	use of appropriate apparatus to record a range of measurements (to include mass, time, volume of liquids and gases, temperature)	
1.2.2 (b)	use of a water bath or electric heater or sand bath for heating	
1.2.2 (d)	use of laboratory apparatus for a variety of experimental techniques including: **(ii)** distillation and heating under reflux, including setting up glassware using retort stand and clamps **(iv)** filtration, including use of fluted filter paper, or filtration under reduced pressure	
1.2.2 (g)	purification of: **(i)** a solid product by recrystallisation	
1.2.2 (k)	safely and carefully handling solids and liquids, including corrosive, irritant, flammable and toxic substances	

Further comment and guidance, including recovery tasks.

Experiment 5 - Qualitative Organic Tests

Preparation Tasks

1) On one side of A4, summarize all of the test for organic functional groups, and the positive results

2) Carry out a risk assessment for your experiment

Introduction

You will carry out a series of qualitative tests for organic functional groups, specifically alkenes, haloalkanes, alcohol, aldehyde and carboxylic acid. Most organic functional groups have a characteristic qualitative test. For example, an alkene can be identified because it reacts with orange/brown bromine solution to produce a colourless solution.

Aims and *skills*

- To identify functional groups in a range of organic compounds
- To use of a water bath or electric heater for heating
- To use laboratory apparatus for qualitative tests for organic functional groups
- To safely handle hazardous substances
- To make and record accurate observations

Equipment

- eye protection
- glass marker pen
- access to distilled or deionised water
- test tubes
- test-tube rack
- dropping pipettes
- beaker (250 cm^3) for water bath
- kettle
- thermometer, 0–110 °C
- spotting tiles/well plates
- spatula or microspatula
- full range pH paper

Risk Assessment

Procedure

Part 1: Identification of an alkene

Label	Identity
heptane	heptane, $C_7H_{16}(l)$
cyclohexane	cyclohexane, $C_6H_{12}(l)$
cyclohexene	cyclohexene, $C_6H_{10}(l)$
$Br_2(aq)$	<0.06 mol dm^{-3} bromine water, $Br_2(aq)$

Procedure

1. For each of the organic substances, in a separate test tube
    a. Add 10 drops of bromine water.
    b. Add one drop of the substance.
    c. Agitate the test tube from side to side to ensure mixing.
    d. Record your observations in a suitable format.

Part 2: Identification of a haloalkane

Label	Identity
1-chlorobutane	1-chlorobutane, $C_4H_9Cl(l)$
1-bromobutane	1-bromobutane, $C_4H_9Br(l)$
1-iodobutane	1-iodobutane, $C_4H_9I(l)$
ethanol	ethanol, $C_2H_5OH(l)$
0.05 mol dm^{-3} $AgNO_3(aq)$	0.05 mol dm^{-3} aqueous silver nitrate(V), $AgNO_3(aq)$

Procedure

1. Set up a small water bath: Half fill a 250cm^3 beaker with warm water (50–60 °C) from a kettle.
2. For each of the haloalkanes, in a separate test tube:
    a. Add five drops of the haloalkane
    b. Add 1 cm^3 ethanol and 1 cm^3 0.05 mol dm^{-3} silver nitrate solution to each tube.
    c. Agitate the test tube from side to side to ensure mixing.
    d. Place the tubes in the water bath for 2–3 minutes.
    e. Record any observations in a suitable format.

Part 3: Distinguishing between primary, secondary and tertiary alcohols

Label	Identity
ethanol	ethanol, $CH_3CH_2OH(l)$
propan-2-ol	propan-2-ol, $CH_3CHOHCH_3(l)$
2-methylpropan-2-ol	2-methylpropan-2-ol, $(CH_3)_3COH(l)$
acidified 0.100 mol dm^{-3} $K_2Cr_2O_7(aq)$	0.100 mol dm^{-3} aqueous potassium dichromate(VI), $K_2Cr_2O_7(aq)$, in 1.4 mol dm^{-3} sulfuric(VI) acid, $H_2SO_4(aq)$

Procedure

1. Using a spotting tile/well plate, add 3 drops of acidified 0.100 mol dm^{-3} potassium dichromate(VI) solution in each of four adjacent wells.
2. Add 2 drops of each alcohol separately to the first three wells – the fourth well is a control to help observe the colour change(s).
3. Observe the colour change(s) over 5-10 minutes.
4. Record any observations in a suitable format.
5. *Disposal instructions: Immediately after your observations have been noted, immerse the spotting tile/well plate in a washing-up bowl of water – this will help minimise any staining of the spotting tile/well plate.*

Part 4: Identification of a carboxylic acid

Chemicals

You are provided with the following:

Label	Identity
0.1 mol dm^{-3} ethanoic acid	0.1 mol dm^{-3} aqueous ethanoic acid, $CH_3CO_2H(aq)$
$Na_2CO_3(s)$	sodium carbonate solid, $Na_2CO_3(s)$

Procedure

1. Separately for 0.1 mol dm^{-3} ethanoic acid and distilled/deionised water
    a. Add 10 drops to a well in a spotting tile/well plate.
    b. Measure the pH of the drop using pH paper.
    c. Add a small spatula measure of sodium carbonate.
    d. Once there is no further sign of reaction, measure the pH of the resultant solution.
2. Record your observations in a suitable format.

Part 5: Identification of an aldehyde

Label	Identity
propanal	propanal, $CH_3CH_2CHO(l)$
propanone	propanone, $CH_3COCH_3(l)$
0.05 mol dm^{-3} $AgNO_3(aq)$	0.05 mol dm^{-3} aqueous silver nitrate(V), $AgNO_3(aq)$
0.4 mol dm^{-3} NaOH(aq)	0.4 mol dm^{-3} aqueous sodium hydroxide, NaOH(aq)
1.0 mol dm^{-3} $NH_3(aq)$	1.0 mol dm^{-3} aqueous ammonia, $NH_3(aq)$

Procedure

1. The first step is to form Tollens' reagent:
    a. Add 2 cm^3 0.05 mol dm^{-3} silver nitrate(V) to a test tube.
    b. Add 2 drops of 0.4 mol dm^{-3} sodium hydroxide solution – a brown precipitate will form.
    c. Add 1.0 mol dm^{-3} ammonia solution dropwise with gentle agitation until the precipitate has just dissolved, to form a colourless solution.
2. Divide the Tollens' reagent between two test tubes.
3. Add 0.5 cm^3 propanal to the first test tube and add 0.5 cm^3 propanone to the second test tube. Gently agitate to ensure mixing.
4. Place the test tubes in a warm water bath (50–60 °C) and leave to stand for 5-10 minutes.
5. Record your observations in a suitable format.
6. **CARE:** Dispose of you reaction mixtures within 30 minutes by rinsing with plenty of water down a foul-water drain.

Student Records

Analysis of your results

1. Part 1 – explain which of the four hydrocarbons are unsaturated. Include the structures of the organic substances formed, where appropriate.
2. Part 2 – explain how this reaction can be used to distinguish between the haloalkanes. Include ionic equations for the reactions involved.
3. Part 3 – explain which of the three alcohols were oxidised. Include structures of the organic substances formed where appropriate.
4. Part 4 – explain which of the two substances were acidic. Include a balanced symbol equation for any reactions that occur.
5. Part 5 – explain which of the carbonyl compounds were oxidised. Include structures of the organic substances formed where appropriate.

Extension Opportunities

1. Explain how far primary, secondary and tertiary alcohols can be distinguished by the use of acidified potassium dichromate(VI). Discuss what further qualitative tests could be carried out to fully distinguish these substances.
2. In Part 2, the diamminesilver(I) ion is formed ($Ag(NH_3)_2^+$), which acts as a mild oxidising agent under alkaline conditions. Predict the reaction product that propanal forms under these conditions. Write half equations and a full redox equation for this reaction (hint: the stoichiometric ratio of aldehyde:hydroxide ion is 1:3).
3. Draw a mechanism for the reaction between cyclohexene and bromine. Ensure that you show the movement of electrons and relevant charges.
4. If you have time, leaving the reaction mixtures in a brightly lit area for 30 minutes. Describe and explain your observations.
5. Use the following bond enthalpies to help interpret your observations for the reactions between haloalkanes and silver nitrate.

Bond	Bond enthalpy / kJ mol^{-1}
C–C*l*	346
C–Br	290
C–I	228

Qualitative Organic Tests – Student Feedback Sheet

Practical skills and techniques covered in this task:		**Awarded**
Use and application of scientific methods and practices		
1.2.1 (b)	safely and correctly use a range of practical equipment and materials, including identification of potential hazards. Learners should understand how to minimise the risks involved.	
1.2.1 (c)	follow written instructions	
1.2.1 (d)	make and record observations/measurements.	
1.2.1 (e)	keep appropriate records of experimental activities	
1.2.1 (f)	present information and data in a scientific way	
1.2.2 (d)	use of laboratory apparatus for a variety of experimental techniques including: **(iii)** qualitative tests for ions and organic functional groups	

Further comment and guidance, including recovery tasks.

Pharmaceutical Poster Task

In this task, you will research information on either Aspirin or Paracetamol, and present your results as a poster. It is important that you use both offline and online sources for your information, and that you correctly cite the sources that you have used, using the Vancouver system of referencing https://www.imperial.ac.uk/media/imperial-college/administration-and-support-services/library/public/vancouver.pdf .

You will have the opportunity to test the melting point of either Aspirin or Paracetamol extracted from tablets, and you will need to include a comparison of this melting point with the published values as part of your work.

Option 1 – Aspirin

Work individually to prepare and make a poster for the wall of the laboratory.

You should include the following:

- the conditions that aspirin helps to relieve or cure, including technical terms such as analgesic, antipyretic and anti-inflammatory;
- the side effects of aspirin, and the alternative treatments for people who are affected by them;
- how aspirin came to be developed over the past 200 years, including the achievements of those responsible for the main developments;
- the chemistry involved in developing the medicine in a usable form;
- the nature and importance of clinical trials.
- The published melting point of aspirin, your experimentally determined melting point of aspirin and explanations of any disparity.

Option 2 – Paracetamol

Paracetamol can be taken in a number of ways and can be bought in many different formulations. Common ones are tablets (500 mg), fizzy dispersible tablets (500 mg), paediatric oral solutions (1 20 mg/5 cm^3), oral suspensions (250 mg/cm and suppositories (1 25 mg). It is also sold in capsules as a mixture with other active ingredients such as codeine and caffeine. Work in individually and explore why there might be advantages in having a number of formulations. Comment on the doses available and suggest a target group for each one. Present your findings as a poster.

You should include the following:

- the conditions that paracetamol helps to relieve or cure, including technical terms such as analgesic, antipyretic and anti-inflammatory;
- the side effects of paracetamol, and the alternative treatments for people who are affected by them;
- the historical development of paracetamol including the achievements of those responsible for the main developments,
- the chemistry involved in developing the medicine in a usable form;
- the nature and importance of clinical trials.
- The published melting point of paracetamol, your experimentally determined melting point and reasons for any disparity.

Useful Hints:

You may find information in reference books, in libraries, in pharmacies and by contacting pharmaceutical companies, or the ABPI (Association of the British Pharmaceutical Industry, 12 Whitehall, London SW1A 2DY, Tel 020 7930 3477 **www.abpi.org.uk**.)

Making a poster

In making a poster the following hints may be useful:

- your poster should be clearly set out, the structure should be clear at a glance;
- people do not like reading a lot of text. Diagrams and flow charts are much easier to take in; text should be readable from at least 2 m;
- explanations should be separate from the main story, perhaps in distinctive boxes;
- the level must be appropriate for the expected audience: you will need to think about what the audience is likely to know already.

You may find information in reference books, in libraries, in pharmacies and by contacting the ABPI (Association of the British Pharmaceutical Industry, 12 Whitehall, London, SWIA 2DY, http://www.abpi.org.uk Tel: 020 7930 3477) or pharmaceutical companies.

Pharmacutical Poster Task – Student Feedback Sheet

Practical skills and techniques covered in this task:		**Awarded**
Use and application of scientific methods and practices		
1.2.1 (d)	make and record observations/measurements.	
1.2.1 (e)	keep appropriate records of experimental activities	
1.2.1 (f)	present information and data in a scientific way	
1.2.1 (g)	use appropriate software and tools to process data, carry out research and report findings	
Research and referencing		
1.2.1 (h)	use online and offline research skills including websites, textbooks and other printed scientific sources of information	
1.2.1 (i)	correctly cite sources of information	
1.2.2 (h)	use of melting point apparatus	