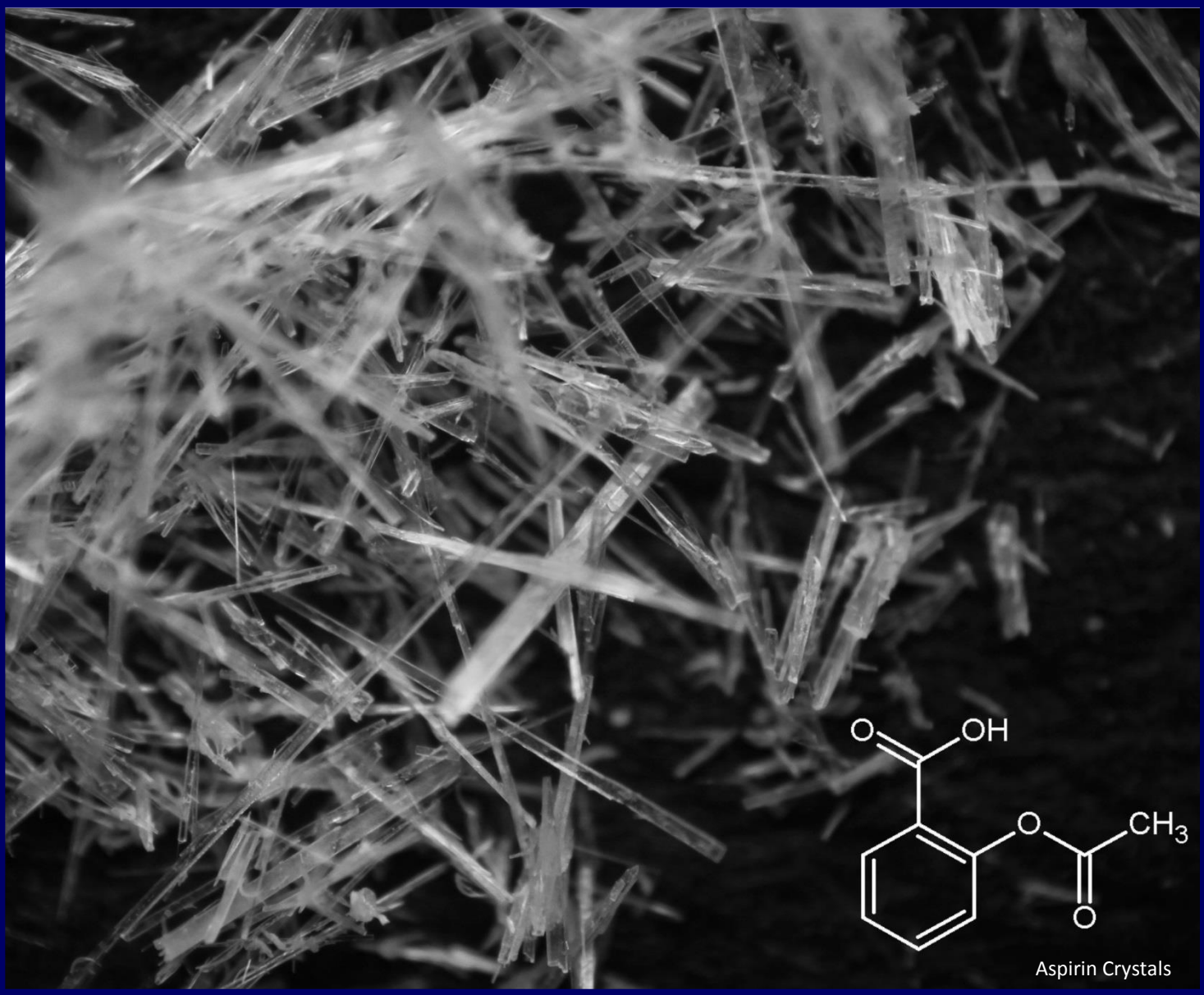

Aspirin Crystals

The Scholars Programme

Killing the Pain:
An Introduction to Organic Synthesis
[Autumn 2015]

Tutor Name

Pupil Name _____

The Scholars Programme – – Pupil Feedback Report

Grade	Marks	What this means
1st	70+	Performing to an excellent standard at first year undergraduate level
2:1	60-69	Performing to a good standard at first year undergraduate level
2:2	50-59	Performing to an excellent standard at A-level
3rd	40-49	Performing to a good standard at A-level
Workings towards a pass	0-39	Workings towards a pass
Did not submit	DNS	No assignment received by The Brilliant Club

Lateness	
Any lateness	10 marks deducted
Plagiarism	
Some plagiarism	10 marks deducted
Moderate plagiarism	20 marks deducted
Extreme plagiarism	Automatic fail

Name of PhD Tutor			
Title of Assignment			
Name of Pupil			
Name of School			
ORIGINAL MARK / 100		**FINAL MARK / 100**	
DEDUCTED MARKS		**FINAL GRADE**	

If marks have been deducted (e.g. late submission, plagiarism) the PhD tutor should give an explanation in this section:

Learning Feedback Comment 1 -	
What you did in relation to this Key Learning Priority	**How you could improve in the future**
Learning Feedback Comment 2 -	
What you did in relation to this Key Learning Priority	**How you could improve in the future**
Learning Feedback Comment 3 -	
What you did in relation to this Key Learning Priority	**How you could improve in the future**
Resilience Comment -	
How you showed learning resilience during the course	**How you could build learning resilience in the future**

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Course Rationale

We all live and breathe organic chemistry. From the chemical reactions taking place within us this very second to the multitude of different fibres present in the clothes we wear and the medications we use to cure illness. Organic chemistry surrounds us everywhere we go. We see it without realising in our skin, the trees and plants outside, even the petrol many use to get to work each day. It's unavoidable and without it we simply would not exist.
Organic chemistry deals with not only the chemistry of life and the natural carbon compounds, but also with the increasing number of synthetic carbon compounds produced in laboratories and factories worldwide. The number of these known compounds today far exceeds 15 million and is increasing by the day as we discover and extract new plant material with potential medicinal properties or create libraries of analogues in a research laboratory.

This course will concern itself with the challenges synthetic chemists and big chemical companies face following the discovery of a new natural product with potential therapeutic applications. How do synthetic organic chemists go about extracting and producing these compounds? How can these chemicals be purified and what is one readily used method for analysing these products?

This course is aimed at getting students interested in practical organic synthesis. They will look at how chemical methods are used within a laboratory to extract active compounds from organic matter and how these active chemicals can then be produced on a larger scale within a laboratory from readily available starting materials. They will be introduced to a variety of commonly used laboratory glassware and equipment as well as learning how to produce an experimental write-up similar to those required at university and used in professional research laboratories.

Mark Scheme

Key Skill	1st	2.1
Communication	☐ Writing is coherent and organised following the logical sequence provided in example. ☐ Experimental method condensed and rewritten in past tense. ☐ Almost faultless spelling, punctuation and grammar. ☐ Writing is of an appropriate style for the scientific document and follows stylistic techniques.	☐ At times the writing is poorly organised and does not follow the logical sequence provided in example. ☐ Experimental method is not written in past tense. ☐ Spelling, punctuation and grammar are weak in places. ☐ Inconsistent use of style throughout a complete document which results in lack of clarity.
Logical Reasoning	☐ There is a clear and structured flow of argument when using logical reasoning to explain how and why specific methods are used in a laboratory. ☐ Arguments and reasoning are expressed with clarity towards a conclusion.	☐ The structure of arguments may be unclear in places and difficult to follow. ☐ There is a lack of clarity within arguments and reasoning towards a conclusion.
Language	☐ Uses appropriate new and scientific vocabulary correctly and effectively ☐ Use of new vocabulary shows a detailed understanding of the concepts covered.	☐ Uses simple new vocabulary effectively but struggles with new vocabulary that is more challenging, in particular spelling of chemical names from formulae. ☐ Scientific vocabulary may be incorrectly used at times.
Attention to Safety Procedures	☐ Show clear understanding and application of the precautions necessary on entering a chemical laboratory. ☐ Chemical hazards have been noted and adhered to. ☐ High levels of intellectual initiative with regard to behaviour in a hazardous laboratory environment.	☐ Show some understanding and application of the precautions necessary on entering a chemical laboratory ☐ Students shows some thought has been taken with regard to risks present. ☐ Good levels of initiative taken with regard to behaviour in a hazardous laboratory environment.
Numerical Analysis	☐ All calculations are clearly structured, clear to follow and correct.	☐ Calculations structures are not always easily followed and contain mistakes in places. ☐ Uses correct formulae although some mistakes are made.

Glossary of Keywords

Acetyl
Acetylation
Alcohol
Aldehyde
Biomass
Carboxylic Acid
CLP Hazard Pictograms
Corrosive
Crude
Distillation
Ester
Ether
Feedstock
Functional Group
Glycoside
Hydrolysis
Ketone
Limiting Reagent
Methoxy
Molar (M)
Molecular Weight (MW)
Natural Product
Organism
Oxidiser
Personal Protective Equipment (PPE)
Pharmacological
Phenol
Recrystallisation
Reflux
Volumetric

Tutorial 1 – What is a Natural Product?

"A chemical substance produced in nature by a living organism that has distinct pharmacological effects" – Webster's Revised Unabridged Dictionary (1913)

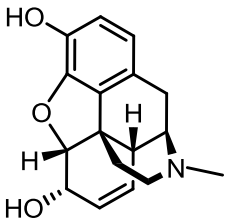
Salix Alba (White Willow) bark, source of salicin (Aspirin)

Papaver somniferum, source of opium (Morphine)

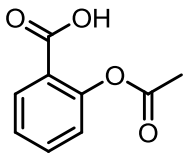
What is the Purpose of Tutorial 1?

- Introduction to the course.
- Understand the usefulness of nature as a source for medicines.
- Understand some simple manipulations that organic chemists use to modify and improve the therapeutic qualities of natural products.
- Understand the common problems of extracting natural products for use in large scale drug synthesis to support a global market.

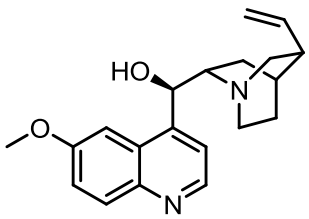
Common Drugs Derived From Natural Sources:



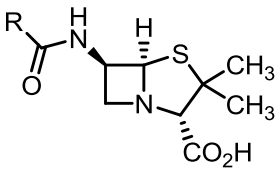
Morphine



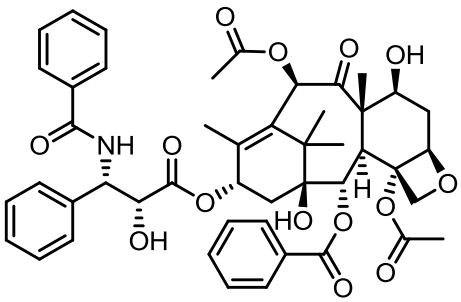
Aspirin



Quinine



Penicillin Core



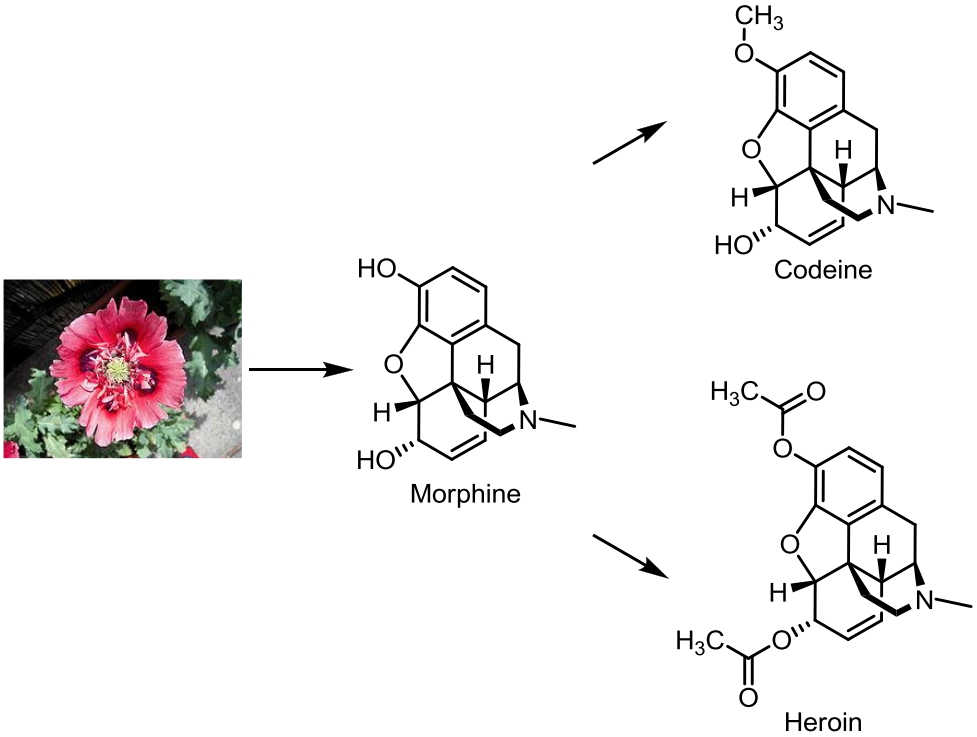
Taxol

Do you know where some of these compounds can be found in nature? Do you know of any other plants with medicinal properties?

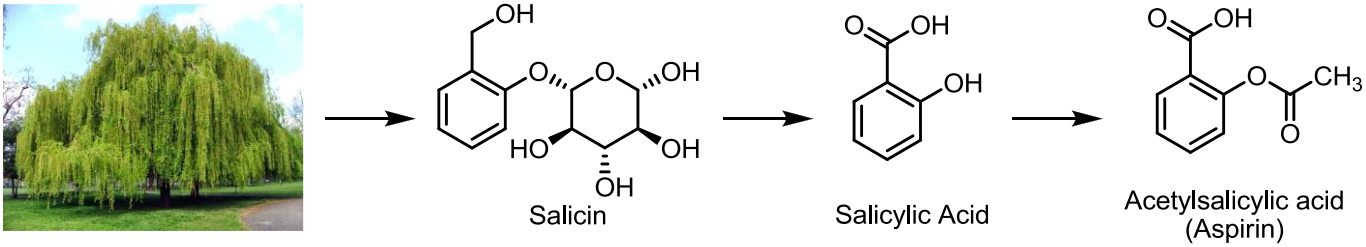
Natural Product to Medicine

What do you notice about the 2 schemes below? What changes have been made when moving from left to right and why do you think these changes might be important? Please **highlight** the changes you can see.

Opium to Morphine



Salicin to Aspirin



Tutorial 1 – Baseline Assessment: The Challenges of Using Plants as Feedstocks for Medicines

Through the use of an **internet search:** Write a short report (500-750 words max) on what you think might be the biggest challenges associated with the use of plants as sources for medicinal compounds. Are the compounds initially extracted always the best ones to use as therapies? What problems do you think there might be of extracting biomass to provide chemical feedstocks to produce drugs on a global scale?

Hints: Search for: the 'History of Aspirin' to begin. How much Aspirin is produced annually for the global market? An approximate yield of Salicin from Willow bark can be found here: http://www.drugs.com/npp/willow-bark.html, what is the significance of this? Do you think plants always produce just a single compound or are they likely to contain mixtures? Are there any factors which might affect the concentrations of compounds found in plant material?

I will expect to see any sources of information included in a reference section (see below for additional guidance) and linked to your text. If you have any questions on this or can't access a specific paper/journal article please ask me. ☺

Referencing Tips and Important Do's and Don'ts

- Referencing is the process of acknowledging the original author or source you have used in writing your essay, review, assignment or any piece of work.
- To reference successfully, you must systematically note the full details of **Author**, **date**, **title and publication details** (journal name, volume, year, page(s)) of the material. For web pages, e-jornals and ebooks, write down the url address and the date accessed.

Reliable sources:

TYPE OF SOURCE	RELIABLE	NOT RELIABLE
WHAT SOMEONE SAID	• Personal communication with an expert on the subject (like an interview with a scientist) • Things mentioned in a documentary	• What my parents, teachers, friends, politicians and not- experts said
PRINTED SOURCES	• Scientific journals and books • Science communication magazines and books • Textbooks • Newspaper articles from serious newspapers: The Independent, The Guardian, The Times etc. • Dissertations or thesis	• Newspaper articles from tabloids like The Daily Mail, The Sun, The Star, The Metro, etc. • Handouts from class

WEB SOURCES	Sites from:
	• Universities (.edu or .ac)
	• Government (.gov)
	• NGO (non-governmental associations): (usually .org, but not all .org sites are associated with NGOs, so pay attention)
	
	• .com or .co sites ONLY if:
	  ○ Sites from academic publishers
	  ○ News agencies (like the BBC)
	  ○ Science websites ONLY if they list authors and have references for their content
	  ○ If they're the website of a corporation, they are a valid source for opinions of the corporation but cannot be taken as objective facts. For example: Monsanto's web page
		• Wikipedia
		• Answers.com, yahoo answers, wiki answers
		• Social Media
		• Blogs
		• Most .com or .co sites, unless they're on the exceptions list to the lift

Tutorial 2 – Safety in the Laboratory and an Introduction to Common Laboratory Equipment

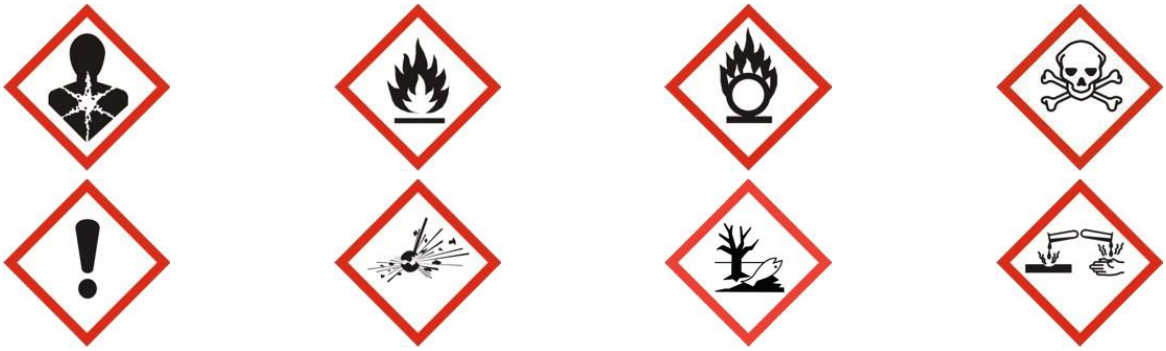
What is the Purpose of Tutorial 2?

- Understanding of the various safety procedures necessary to create a safe working environment.
- Introduction to a variety of common laboratory equipment and their uses.

Laboratory Safety Quiz

What do you already know about lab safety? Complete the quiz and we will discuss and go through together

1. When working in the laboratory with chemicals, what personal protective equipment (PPE) should be worn at all times?
   - **a.** Always wear goggles and lab coats. Gloves should also be worn when needed
   - **b.** Goggles only
   - **c.** None of the above
2. Can open-toed or open-heeled shoes be worn in the lab?
   - **a.** Yes
   - **b.** Never
   - **c.** May be
3. Is it permissible to have any drink or food in the laboratory?
   - **a.** Yes
   - **b.** Never! There is no eating and no drinking permitted in the labs.
   - **c.** Water only
4. Do you know what each of the CLP hazard pictograms below mean?



5. When diluting an acid with water, always remember to:
   - **a.** Pour water into acid
   - **b.** Pour acid into water
   - **c.** Add both at the same time
6. Where in the laboratory should waste chemicals be disposed of properly?
   - **a.** Garbage
   - **b.** Sink
   - **c.** Dispose of in the appropriately labelled container
7. In the event a chemical spill (such as an acid or base gets into your eyes or on your skin), what should you do?
   - **a.** Immediately rinse affected area in running water for 15 minutes
   - **b.** Report to the police
   - **c.** Run to get the school nurse
8. Describe the procedure for smelling a chemical
   - **a.** Stick your nose close to the vessel and breathe deeply
   - **b.** Point container away from face while gently fanning vapours towards nose
   - **c.** Never smell a chemical

Common Laboratory Equipment

Below is a table with commonly found items of laboratory equipment, which ones have you already come across in your practical lessons? What is each item used for?

Reflux condenser			Electronic balance		
Beaker			Measuring cylinder		
Buchner flask and Hirsch Funnel			Volumetric pipette		
Fume cupboard			Hot water bath		
Spatula			Filter paper		
Stand and clamp			Conical flask		
Test-tube			Pear-shaped flask		
Test-tube rack			Round-bottom flask		
Bunsen Burner			Thermometer		
Dropping Pipette			Burette		

Practical Organic Chemistry

General Safety Details

Before you do anything in the laboratory you MUST read the following safety details:

1. Safety glasses or goggles MUST be worn at all times in the laboratory. You MUST also wear a lab coat. You should use disposable gloves when handling toxic or corrosive substances.

2. Be sure you know the whereabouts of the nearest fire extinguisher, sand bucket, fire blanket, eye bath, and first-aid kit. Many organic substances are both volatile and flammable and hence constitute a fire hazard.

3. Wet and dirty benches are a safety hazard. Several of the compounds used in this course are toxic and may be absorbed by the skin. Organic solvents can break down the fat barriers on the skin and facilitate the absorption of toxic substances. A number of the compounds are skin irritants. If you get chemicals on your skin, rinse them off as soon as ASAP with water. Take care in handling strongly acidic solutions. Use a funnel where appropriate and rinse it immediately after use. You MUST read the risk assessment for each practical before you start work.

4. Clear up apparatus at the end of an experiment and dispose of residues as instructed for the particular practical. Do not leave dirty apparatus out on the bench.

5. If you break glassware, sweep up the broken pieces and put them in the broken glass bin. Avoid handling sharp pieces of glass. If you chip the glassware bring it to the technician to check that it is still safe to use.

6. NO CHEMICALS, SAMPLES OR APPARATUS MAY BE REMOVED FROM THE LABORATORY.

7. Experimental work may only be carried out when the demonstrator is on duty. Attendance at any pre-practical instruction sessions is compulsory. Students, who do not attend this, may not be allowed to carry out the practical.

8. No food or drink may be consumed in the laboratory.

9. If you suffer from any disability or health problem (e.g. Physical disability, asthma or skin allergy) which might be exacerbated by a practical or preclude you from safely carrying out an experiment, you must inform the demonstrator before starting the practical so that appropriate assistance can be given.

10. Be sure that you understand the chemistry and potential hazards involved in an experiment before you starts work. If in doubt ASK. Plan your work so that it can be safely completed within the time allocated. On some days it may be necessary to complete an experiment in the following session (e.g. to carry out a melting point (MP.) on dry material.

Please sign to show you have read the above _____

General Details

Practical handbooks and samples will be marked during the course. The timetable for handing in handbooks will be given in the pre-lab sessions. It is your responsibility to ensure that your work is marked as these marks will contribute to your final grade.

Organic compounds are known by both their systematic and trivial names. The systematic names are structure based. The trivial names are accepted by IUPAC and in some cases are the preferred names. They are used in research, in industry and in international scientific literature. They are also the names that are used by the suppliers and are on the bottles. Hence, for safety reasons, we will use them. However, it is important to remember the systematic naming of organic compounds because this is used for more complicated structures

Guidelines for Writing Practical Reports

Practical work should be written up at the time at which you are doing the experiment. It should be written up in your handbook in the space provided. Each experiment should be given a title and the date included at top of page. A short introductory section may set out the objectives of the experiment, the formulae of the substances involved and any hazards. I would recommend using a table to show the chemicals used and amounts (in moles) and equivalents (based on moles to determine the limiting reagents).

The practical record should contain a clear, brief statement of what you did, the materials, their quantities, the apparatus and the techniques you used. You should report what you observed, including any colour changes, gas or heat evolution or formation of a precipitate.

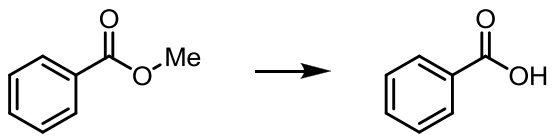
The report should be written in the past passive tense. [E.g. Piperonal (1.00 g) was dissolved in ethanol ($10cm^3$) in a conical flask.] Whilst being concise, the report should be sufficiently detailed for the experiment to be repeated without recourse to other information. Each step may be numbered. A typical report (including the formulae etc.) for this course would occupy about 1-2 pages max.

You should report the yield (in g and % theoretical where appropriate) of the products together with their physical constants (melting point (MP.)) and appearance. Where possible a comparison with the literature data should be reported together with a reference to the source of information. When quoting the percentage yield remember to indicate the basis on which it was calculated. In presenting any calculation make sure that the steps are clearly given so the progression to the final figure may be checked.

Calculation of Yield

The percentage yield is calculated in terms of the 'limiting' reagent (or starting material) and refers to the amount of dry, pure material presented at the end of the experiment available for further use. It may be calculated as in the following example:

Reaction



Mr 136 Mr 122

Quantity used	Final quantity obtained
1g	0.5g

Theoretical yield: 122/136 x 1g = 0.9g

Percentage yield: 0.5/0.9 x 100 = 55% (based on methyl benzoate)

In quoting the yield care must be taken to use only the appropriate number of significant figures bearing in mind the accuracy of the initial and final weighing.

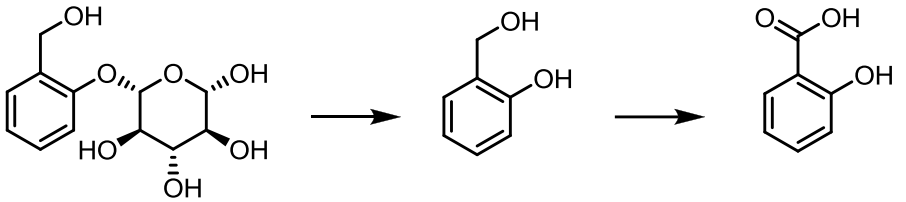
Marking Practical Work

In marking practical work, marks will be awarded according to the guidelines set out in the mark scheme on page5 and:

- The quality and accuracy of the written work, observations and data.
- The presentation of written work including date, title, formulae and the correct spelling of chemical names (the incorrect spelling of chemical names can present a safety hazard).
- The answers to the questions or problems.
- The quality and quantity of the sample that has been prepared may also be assessed.

Penalties will be awarded for submission of late work.

Tutorial 3 – Extraction of an Active Chemical from Willow Bark (Adapted from Salters Resources)



Salicin (From Bark) → Saligenin → Salicylic Acid

What is the Purpose of Tutorial 3?

- Introduction to the practical laboratory.
- Introduction to setup of common laboratory apparatus and a use of a common test for the phenol group of active compounds
- Introduce a method for testing for presence for phenol functional group.

Hazard Assessment

- **Sulfuric acid** can cause burns. It gives off an irritating vapour that can damage the eyes and lungs.
- **Ethanol** is flammable.
- **Potassium manganate(vii)** is harmful and an oxidising agent
- **2-Hydroxybenzoic acid (salicylic acid)** is harmful by ingestion and is irritating to the skin and eyes.

Procedure

Set up apparatus suitable for heating approximately 30 cm^3 of reaction mixture using a water bath. The reaction is quite slow. You will need to heat the mixture for 15 minutes using a water bath. Use a condenser to prevent any volatile liquids escaping. **Get your apparatus checked by the demonstrator before you start the reaction.**

1. Take a sample of willow twigs or bark and chop into fine pieces using a coffee grinder/scissors or scalpel

2. Place 2 g of the finely chopped pieces into your flask.

3. Cover with approximately 25 cm^3 of a mixture containing equal parts of the 2 mol dm^{-3} sulphuric acid and 0.2 mol dm^{-3} potassium manganate(vii) solution.

In the body the willow extract is first hydrolysed by enzymes in the stomach. The resulting substance is oxidised in the bloodstream to become the active chemical, which is the drug.
Which chemical in this experiment do you think causes the hydrolysis and which causes the oxidation?

4. Reflux the mixture for 15 mins. Note any change in the appearance of the mixture in the flask. Allow the mixture to cool slightly.

5. Use a dropping pipette to transfer about 1 cm^3 of the reaction mixture from the flask to a test tube.

6. Set up 3 test tubes so that each contains approximately 1cm^3 of a different concentration of the salicylic acid solution (0.2 g per 100 cm^3/0.02 g per 100 cm^3/0.002 g per 100 cm^3)

7. Add 1 cm^3 of the 0.1 mol dm^{-3} neutral iron (lll) chloride solution to each of the 4 test tubes. Record your observations in a table.

Results

Include the answers to the following questions in your write up.
1. What do your results tell you about the sensitivity of the Iron (lll) chloride (ferric chloride) test?
2. What do your results tell you about the composition of the willow extract?

This page is for writing your report. I have added sections to help you this time and use as template / guide in future.

Title: Date:

Introduction / Purpose of Experiment:

Scheme:

Hazards:

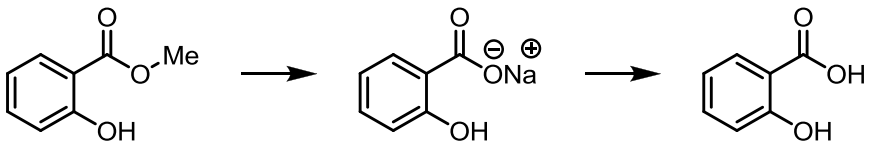
Procedure: (written in past passive tense include any observations to e.g. colour changes to solutions etc.)

1. Sample of bark chopped using a coffee grinder and fine pieces (g) placed in pear-shaped flask (50 cm^3)
2.

Results Table:

Questions:

Tutorial 4 – Preparation of 2-hydroxybenzoic acid (Adapted from RSC Resources)



Mr 152.15 Mr 160.10 Mr 138.12

What is the Purpose of Tutorial 4?

- To produce 2-hydroxybenzoic acid from oil of wintergreen.
- Introduce students to a hydrolysis reaction with aqueous sodium hydroxide that will produce sodium 2-hydroxybenzoate.
- Convert sodium 2-hydroxybenzoate salt into the desired carboxylic acid product with hydrochloric acid.

Many organic compounds are found in plants. 2-Hydroxybenzoic acid (salicylic acid) can be made from methyl 2-hydroxybenzoate which is obtained as oil of wintergreen by distillation from the leaves of Gaultheriae procunbers. Oil of wintergreen is 98% methyl 2-hydroxybenzoate.

Hazard Assessment

- **Hydrochloric acid** can cause burns. It gives off an irritating vapour that can damage the eyes and lungs.
- **Methanol** is flammable.
- **Sodium hydroxide** can cause severe burns to the skin and is dangerous to the eyes.
- **2-Hydroxybenzoic acid (salicylic acid)** is harmful by ingestion and is irritating to the skin and eyes.

Procedure

Set up apparatus suitable for heating approximately 30 cm^3 of reaction mixture using a water bath. The reaction is quite slow. You will need to heat the mixture for 30 minutes using a water bath. Use a condenser to prevent any volatile liquids escaping. **Get your apparatus checked by the demonstrator before you start the reaction.**

1. Put 2 g of oil of wintergreen (methyl salicylate) into your flask and add 2 cm^3 of 2 mol dm^{-3} sodium hydroxide (CARE!). Aqueous sodium hydroxide is particularly prone to bumping so you will need some anti-bumping granules. Then heat over a boiling water bath for 30 minutes.

While the mixture cools make a list of the possible compounds present in the mixture.
Why do we use a water bath to heat instead of a naked flame?

2. Pour the mixture into a small beaker surrounded by a mixture of ice and water. Add concentrated hydrochloric acid (CARE!) to the mixture dropwise until it is just acidic (check with indicator paper), stirring all the time.

Why do you need to keep the mixture cool during this process?

3. Filter the product under vacuum (Buchner flask and Hirsch funnel and suction apparatus). Wash the product with a little ice cold water and transfer it to a weighed watch glass. Allow to dry overnight.

Results

Include the answers to the following questions in your write up.
1. How can you tell from observing the process that a new substance has been formed in the reaction?
2. What has happened to the methanol formed in the reaction?
3. What is oil of wintergreen used for nowadays?

Remember: Include the mass of product formed from 2 g of oil of wintergreen and percentage yield in your write up!

This page is for writing your report. Add to the given sections below to produce a complete report

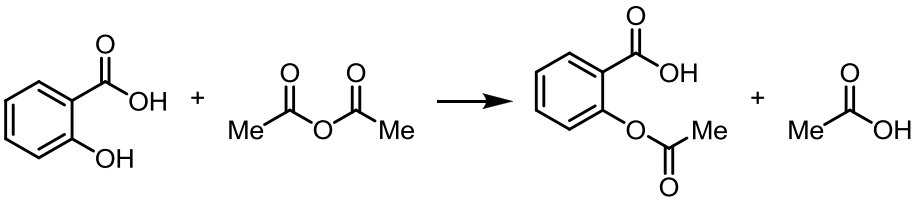
Reagents Table: (Complete tables like this to easily see amounts of reagents needed at a glance)

Reagent	mmol	mw	Mass (g)	Density (g/cm^3)	volume (cm^3)	Hazards
methyl salicylate		152.15	2.00	1.17		
$NaOH_{(aq)}$ (2M)					2.0	

Tutorial 5 – The Preparation of Aspirin (Adapted From RSC Resources)

What is the Purpose of Tutorial 5?

- Use previously produced 2-hydroxybenzoic acid and react with ethanoic anhydride to make aspirin
- Purify crude aspirin product through technique of recrystallisation.
- Produce a full laboratory report including percentage yields and MP.



Mr 138.12 Mr 102.09 Mr 180.16 Mr 60.05

Hazard Assessment

- **Ethanoic anhydride** is flammable and causes burns.
- **2-Hydroxybenzoic acid (salicylic acid)** is harmful by ingestion and is irritating to the skin and eyes.
- **Phenol** is toxic by ingestion and skin absorption. It can cause severe burns. Take care when removing phenol from the bottle because the solid crystals can be hard to break up.
- **Phosphoric acid** is irritating to the eye and causes burns

Procedure

The reaction takes place easily in acidic solution but the product is formed as part of a mixture containing several other compounds.
Note that ethanoic anhydride reacts readily with water so all the apparatus must be dry.

Set up apparatus suitable for heating approximately 30 cm^3 of reaction mixture using a water bath. The reaction is quite slow. You will need to heat the mixture for 30 minutes using a water bath. Use a condenser to prevent any volatile liquids escaping. **Get your apparatus checked by the demonstrator before you start the reaction.**

1. Take about 1 g of your sample of 2-hydroxybenzoic acid and weigh it accurately. Put it into a dry pear shaped flask and add 2 cm^3 of ethanoic anhydride followed by 8 drops of concentrated phosphoric acid. Put a condenser on the flask. In a fume cupboard, warm the mixture in a hot water bath, with swirling, until all the solid has dissolved and then warm for another 5 minutes.

2. Carefully add 5 cm^3 of cold water to the solution. Stand the flask in a bath of iced water until precipitation appears to be complete. It may be necessary to stir vigorously with a glass rod to start the precipitation process.

3. Filter the product using vacuum filtration. Wash the product with a little ice cold water and allow to dry by sucking air through continuously for another 5-10 mins.

Purification by Recrystallisation

When an organic compound has been made it needs to be purified, particularly if it is a pharmaceutical chemical. This is because most organic reactions produce by-products but, even if the reaction is a 'clean' one, the purity standards for many products are so stringent that small amounts of other compounds have to be removed.

In the laboratory, this is often done by recrystallisation. The general method is to find a solvent that dissolves the product more readily at high temperature than at low temperature, make a hot solution, and allow to crystallise on cooling. The crude product might contain;

- Impurities which are insoluble in the solvent;
- Impurities which are slightly soluble in the solvent; and
- Impurities which dissolve readily in the solvent.

Procedure

1. Dissolve your crude product in about 10 cm^3 of ethanol in a 50 cm^3 conical flask and warm in a water bath to dissolve the solid.
2. If any solid material remains undissolved, filter the solution under vacuum and discard residue.
3. Add 25 cm^3 of warm water (about 50 °C) to the clear alcohol solution. If any crystals appear at this point, heat the contents of the flask until they dissolve.
4. Set the flask aside to cool, observing it carefully.
5. When crystals start to form, cool the flask by placing it in ice. The crystallisation process will then go to completion.
6. Collect the crystals by vacuum filtration (Buchner flask and Hirsch funnel and suction apparatus).
7. Allow the crystals to dry overnight in a desiccator or overnight in an oven at 50 °C.
8. Come back tomorrow to do a melting point to determine purity.

Melting Point

Measuring the melting point of a substance is a good way to test for purity. In this experiment you use the melting point as a way of investigating the purity and identity of laboratory prepared aspirin samples.

A pure substance usually has a sharp melting point – i.e. a narrow temperature range during which it changes from a solid to a liquid. A substance which contains impurities often melts over a range of several degrees.
Any impurities in the substance cause a lowering and broadening of this characteristic temperature.

Substance	Melting Point (MP.)
2-hydroxybenzoic acid	158-160 °C
Aspirin	138-140 °C

Method

1. If you do not have sealed melting point tubes, heat the end of a capillary tube in a Bunsen burner flame until the glass softens and the end is sealed. Do not heat the tube so strongly that it bends. Leave it on a heatproof mat to cool.
2. Fill the melting point tube to a depth of about 0.5 cm with dry impure aspirin sample
3. Seal a second tube and fill it to a depth of about 0.5 cm with dry crystals of your purified aspirin.
4. Place each tube in the melting point apparatus; slowly increase the temperature and note the temperature range over which the substances melt.

Tutorial 5 – Final Assessment:

Using the knowledge you have gained in the course so far, produce a full and complete experimental report. Include in your report the answers to the questions below:

1. How might material be lost at each stage (in reaction and recrystallisation)?
2. How could losses be minimised?
3. Why might the apparent yield be too large?
4. On the basis of melting point is it reasonable to conclude that the substances tested contain aspirin?
5. Account for any difference between the melting points of the crude and recrystallised samples of aspirin.
6. What other impurities could there be in the aspirin made in the laboratory?
7. Describe in molecular terms your ideas of what happens when a substance melts.
8. Why does this explanation support the fact that aspirin has a lower melting point than 2-hydroxybenzoic acid?
9. What is the theoretical yield of product (aspirin) from 2 g of oil of wintergreen?
10. Calculate overall percentage yield from oil of wintergreen (based on your own results).
11. Based on Q10, do you think this is a good method for producing aspirin? (Justify your answer)

Tutorial 6 – Feedback

What is the Purpose of Tutorial 6?

- Reflect on skills learned on the programme with a view to encouraging resilience
- Receive feedback on final assignment

What are the key questions?

- What strengths have I demonstrated in my work and what areas of development are there left for me to address?
- What steps do I need to take to improve my academic output?
-

Activity 2 – Marking your own

Using the mark scheme, identify three things you did well and three things you could improve.

What went well?

1.

2.

3.

What could be improved?

1.

2.

3.

Activity 3 – Responding to feedback

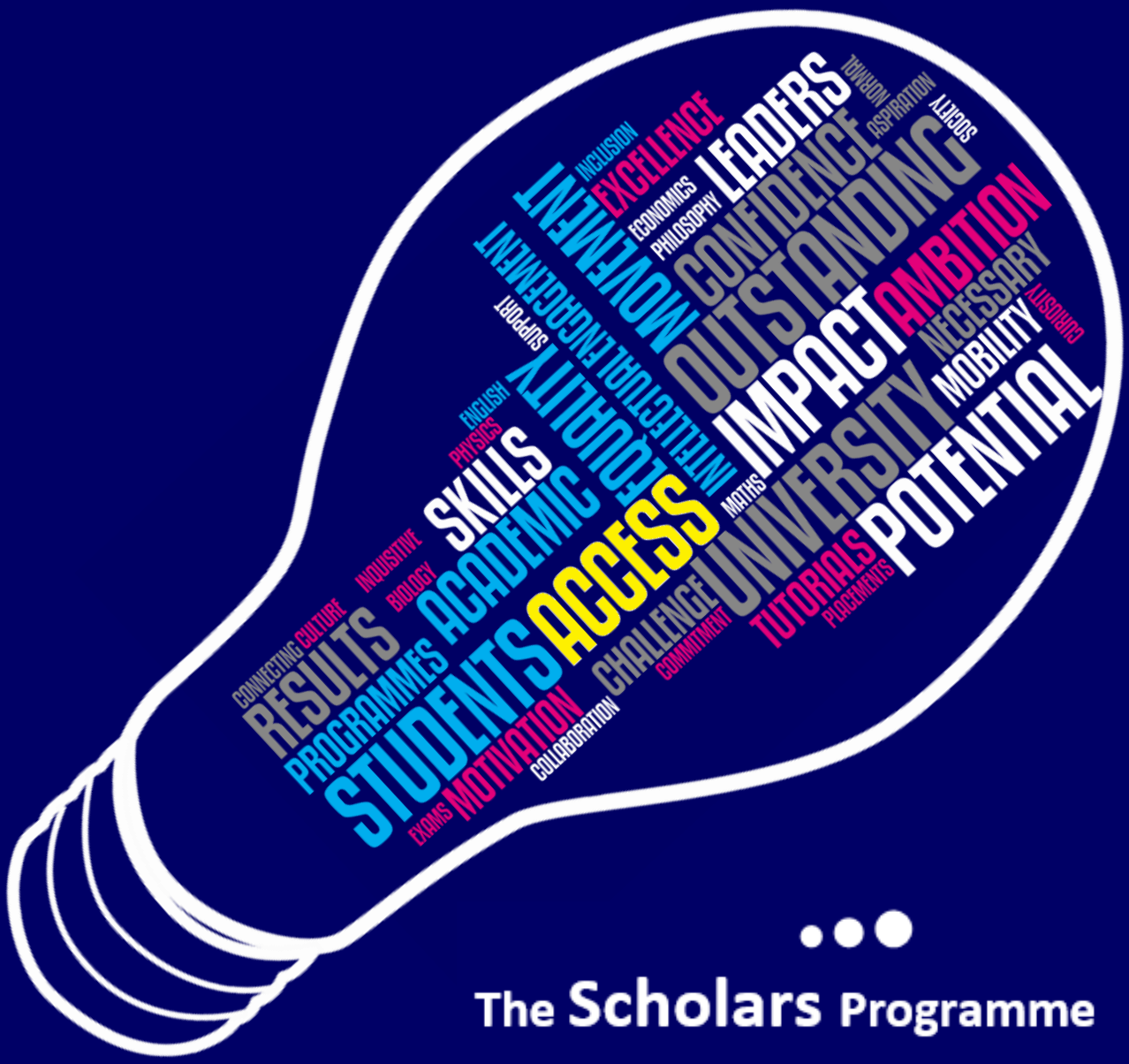
Read through your feedback sheet and highlight on your assignment where you received positive feedback (one colour) and areas for improvement (another colour). You might find it helpful to annotate this with a brief comment or two.

(Acknowledgement: The structure and the activities in this feedback tutorial were first designed by Hannah Manktelow in January 2015. We have repeated them here as they demonstrate thoughtful and excellent planning, and can be used by all tutors to ensure a successful and productive feedback tutorial.)

University Applications Guidance

For course choices and careers information:	www.brightknowledge.org.uk
For information and statistics on universities and course:	www.unistats.direct.gov.uk
For comprehensive information on universities, including rankings:	www.thecompleteuniversityguide.co.uk

PLEASE NOTE – The Scholars Programme is designed to support the university applications process at your school. The work you complete for the programme should add to your schoolwork rather than detract from it. Please be aware of deadlines and try to manage your workload appropriately. If you are worried about the Scholars Programme interfering with your schoolwork then please speak to your teacher.



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