Investigations Folio:
Organic Preparation – Ester Hydrolysis

Background Information

- Ester hydrolysis is the reverse reaction of esterification. Esters are hydrolysed very slowly by water but much more quickly by hydroxide ions from sodium (or potassium) hydroxide. The process of alkaline hydrolysis of an ester is sometimes called saponification because of its use in soap manufacture.
- In this practical, you are going to hydrolyse the ester methyl salicylate and separate the alcohol and carboxylic acid products.

Assessment Conditions

- You will have the 110 minute Tutorial to complete the hydrolysis, distillation and filtration **under direct supervision**.
- During this period you will be assessed in your ability to follow the provided instructions in the selection and manipulation of apparatus and the implementation of safe procedures.
- You will also be required to use the spaces provided to record annotated evidence of your observations and understanding of the procedure (in the format of a flowchart) – **to be submitted during the investigation under direct supervision**.
- You will required to elaborate upon this evidence in the construction of a final report. **No new evidence may be introduced in the final submission**. You will have one further week to complete this report.
- You will work collaboratively, in pairs, during the investigation, but **each student must demonstrate original thought through an individual report**.

Aims

- To hydrolyse a sample of the ester methyl salicylate.
- To remove the alcohol product by distillation.
- To separate the carboxylic acid product.

Apparatus & Reagents

Test tubes	Methyl salicylate (oil of wintergreen)
100-150 mL flask	Concentrated hydrochloric acid
Boiling chips	2 mol.L^{-1} sodium hydroxide solution
Suction filter system (or filter paper and funnel)	1 mol.L^{-1} potassium dichromate solution
Thermometer	Litmus paper
Distillation apparatus	**Safety glasses**

Safety Notes

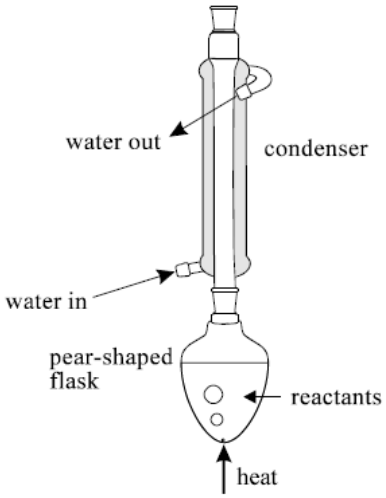
Consult the appropriate MSDS for each of the chemicals prior to performing the practical.

- Using the MSDS identify the hazards associated with the use of sodium hydroxide and hydrochloric acid and state how you will respond to the identified hazards during the practical investigation. [IAE1]

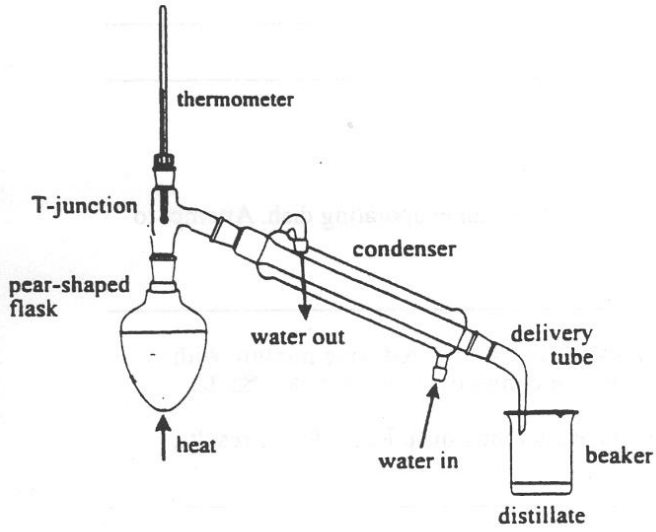
Hazard	Response

Instructions

- Place 2 mL of methyl salicylate in a pear shaped distillation flask. Add 25 mL of 2 mol L^{-1} sodium hydroxide solution and one or two boiling chips. Reflux the mixture gently for about 30 minutes, by which time any oily drops of the ester should have disappeared.



- Distil the mixture slowly to collect about 1 mL of distillate. Keep this for testing. Record the temperature range at which you collected the distillate.



- Add concentrated hydrochloric acid to the cooled residual solution, with constant stirring, until the solution is just acid to litmus. Filter off the crystals under suction and wash with water.
- Dry the crystals thoroughly and record your observations.
- Test a small portion of the distillate with a few drops of acidified potassium dichromate and warm. Record your observations.

Safety

WORK-PRACTICE DATA SHEET - THIS IS A SUMMARY ONLY - FULL REPORT AVAILABLE

Chemwatch 1823 - HAZARDOUS SUBSTANCE. DANGEROUS GOODS. According to GHS (Model WHS), and the ADG Code. - Company Contact No. 1800 039 008 (24 hours)

SODIUM HYDROXIDE

INGREDIENTS	CAS No	%	TWA
sodium hydroxide	1310-73-2	>98	

UN No: 1823
Hazchem Code: 2W
DG Class: 8
Subsidiary Risk: None
Packing group: II
Poisons Schedule: S6 NZS3
Max Haz Rating:: 4 (red)

PROPERTIES

Solid.
Mixes with water.
Corrosive.
Alkaline.
Does not burn.

HAZARD STATEMENT

May be corrosive to metals.
Causes severe skin burns and eye damage.
Causes serious eye damage.

SAFE STORAGE WITH OTHER CLASSIFIED CHEMICALS

X: *Must not be stored together*
0: *May be stored together with specific preventions*
+: *May be stored together*

PRECAUTIONS FOR USE

Engineering Controls:
Local Exhaust Ventilation recommended.
Glasses:
Chemical goggles.
Full face- shield.
Gloves:
1.BUTYL 2.NAT+NEOPR+NITRILE
Respirator:
Particulate
Storage & Transport:
Keep locked up.
Store in cool, dry, protected area.
Restrictions on Storage apply. Refer to Full Report.
Fire/Explosion Hazard:
Vapours/ gas heavier than air.
Toxic smoke/fumes in a fire.
Environment:
This material and its container must be disposed of as hazardous waste.

EMERGENCY

FIRST AID
Swallowed:
Give water (if conscious). URGENT MEDICAL ATTENTION.

EMERGENCY

Eye:
Wash with running water (15 mins). Medical attention.
Skin:
Flood body with water. Remove contaminated clothing.
Wash with water & soap. MEDICAL ATTENTION.
Inhaled:
Fresh air. Rest, keep warm. If breathing shallow, give oxygen. Medical attention.
Advice to Doctor:
Supportive care.
Fire Fighting:
Keep surrounding area cool. Water spray/fog.
Spills & Disposal:
Avoid dust.
Prevent from entering drains.
Contain spillage by any means.
Sweep/shovel to safe place.
Take off immediately all contaminated clothing.
To clean the floor and all objects contaminated by this material, use water.

Results

Record observations for each of the stages of the process and explain their significance.

	Observations	Explanation
Reflux:	Before: During: After:	
Distillation:		
Acidified dichromate:		
Acidification of solution:		

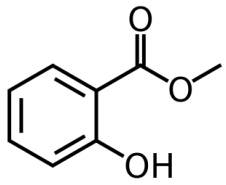
[IAE2, KA2]

Record the boiling range of your distillate _____ [IAE2]

Teacher's Signature: _____

Analysis/Discussion

The structural formula of methyl salicylate is:



Using the structure above, construct a detailed flow diagram for the entire process including the following:

- All reagents used
- Appropriate equations (balanced where possible)
- Reactions conditions

[IAE1, KA2, KA4]

Respond to and incorporate the following questions into your analysis/discussion:

- The general equation for the formation of esters is shown below:

$$\text{carboxylic acid} + \text{alcohol} \overset{\text{sulfuric acid}}{\rightleftharpoons} \text{ester} + \text{other product}$$

  Name the other product formed in the reaction.

  _____ [KA1]

  State the function of the sulfuric acid.

  _____ [KA1]

- Explain the purpose of heating under reflux in ester formation and hydrolysis.

  _____

  _____

  _____

  _____ [IAE4]

- State the function of the boiling chips that are used in the reflux and distillation.

  _____

  _____ [KA1]

- Explain the purpose of adding HCl to the residual solution during the investigation.

  _____

  _____

  _____

  _____ [IAE4]

- Explain why salicylic acid is insoluble in water

  _____

  _____

  _____

  _____

  _____

  _____ [KA2]

Investigations Folio:
Organic Preparation – Ester Hydrolysis

SACE Number/Name: _____

		Intended Student Learning	Knowledge & Application	Investigation, Analysis & Evaluation
Procedures	Follow instructions accurately to prepare organic compounds using reflux, distillation, and extraction.			
Safety & Ethics	Recognise hazards and work safely during a practical investigation.			
Information & Data	In investigations, make and record careful and honest observations and measurements.			
Interpretation & Evaluation	Analyse and evaluate procedures from an investigation, and indicate the effect on the results. ▪ Flowchart ▪ Esters ▪ Reflux ▪ Acidification ▪ Solubility			
Communication	Communicate knowledge and understanding of chemistry in the required format. Write appropriate chemical equations.			
	Evidence of Learning – Distribution			
	Grade by Subdivision			

	A	B	C	D	E
Investigation, Analysis, and Evaluation	Designs a logical, coherent, and detailed chemistry investigation. Obtains records, and represents data, using appropriate conventions and formats accurately and highly effectively. Systematically analyses and interprets data and evidence to formulate logical conclusions with detailed justification. Critically and logically evaluates procedures and discusses their effect on data.	Designs a well-considered and clear chemistry investigation. Obtains, records, and represents data, using appropriate conventions and formats mostly accurately and effectively. Logically analyses and interprets data and evidence to formulate suitable conclusions with reasonable justification. Logically evaluates procedures and their effect on data.	Designs a considered and generally clear chemistry investigation. Obtains, records, and represents data, using generally appropriate conventions and formats with some errors but generally accurately and effectively. Undertakes some analysis and interpretation of data and evidence to formulate generally appropriate conclusions with some justification. Evaluates procedures and some of their effect on data.	Prepares the outline of a chemistry investigation. Obtains, records, and represents data, using conventions and formats inconsistently, with occasional accuracy and effectiveness. Describes data and undertakes some basic interpretation to formulate a basic conclusion. Attempts to evaluate procedures or suggest an effect on data.	Identifies a simple procedure for a chemistry investigation. Attempts to record and represent some data with limited accuracy or effectiveness. Attempts to describe results and/or interpret data to formulate a basic conclusion. Acknowledges that procedures affect data.
Knowledge and Application	Demonstrates deep and broad knowledge and understanding of a range of chemical concepts. Develops and applies chemical concepts highly effectively in new and familiar contexts. Critically explores and understands in depth the interaction between science and society. Communicates knowledge and understanding of chemistry coherently, with highly effective use of appropriate terms, conventions, and representations.	Demonstrates some depth and breadth of knowledge and understanding of a range of chemical concepts. Develops and applies chemical concepts mostly effectively in new and familiar contexts. Logically explores and understands in some depth the interaction between science and society. Communicates knowledge and understanding of chemistry mostly coherently, with effective use of appropriate terms, conventions, and representations.	Demonstrates knowledge and understanding of a general range of chemical concepts. Develops and applies chemical concepts generally effectively in new or familiar contexts. Explores and understands aspects of the interaction between science and society. Communicates knowledge and understanding of chemistry generally effectively, using some appropriate terms, conventions, and representations.	Demonstrates some basic knowledge and partial understanding of chemical concepts. Develops and applies some chemical concepts in familiar contexts. Partially explores and recognises aspects of the interaction between science and society. Communicates basic chemical information, using some appropriate terms, conventions, and/or representations.	Demonstrates limited recognition and awareness of chemical concepts. Attempts to develop and apply chemical concepts in familiar contexts. Attempts to explore and identify an aspect of the interaction between science and society. Attempts to communicate information about chemistry.