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Organic Chemistry

Microscience Experiments

Teaching and Learning Materials

MANUAL FOR LEARNERS

Compiled by Beverly Bell & Christopher Gunter
Edited by Prof. JD Bradley and Jane Spriggs

The UNESCO-Associated Centre for Microscience Experiments
RADMASTE Centre
University of the Witwatersrand, Johannesburg

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UNITED NATIONS EDUCATIONAL, SCIENTIFIC AND CULTURAL ORGANIZATION

INTERNATIONAL ORGANIZATION FOR CHEMICAL SCIENCES IN DEVELOPMENT

INTERNATIONAL UNION OF PURE AND APPLIED CHEMISTRY – IUPAC

IN COLLABORATION WITH

THE UNESCO-ASSOCIATED CENTRE FOR MICROSCIENCE EXPERIMENTS
THE RADMASTE CENTRE
UNIVERSITY OF THE WITWATERSRAND
JOHANNESBURG, SOUTH AFRICA

THE INTERNATIONAL FOUNDATION FOR SCIENCE EDUCATION
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Organic Chemistry Microscience Experiments

FOREWORD

The International Organisation for Chemical Sciences in Development (IOCD) was established more than 22 years ago. Since its inception, it has been assisting its partners in many different parts of the world - with special emphasis on the developing countries - to create and develop projects on chemical sciences. Professor Glen Seaborg and I, together with all our colleagues, have always tried to find the best solutions during the development stages of such projects. We have worked with the different Intergovernmental (IGO) and Non-governmental (NGO) organisations, and especially with UNESCO - the unique organisation of the United Nations system with the international mandate to develop programme activities within the basic science disciplines and mathematics. Amongst these the chemical sciences have been described as the "essential science" by Professor Glen Seaborg.

Today we are very pleased to present to the world community, the new teaching and learning packages on Organic Chemistry Microscience Experiments. These materials can be considered as an extension of the previous Advanced Teaching and Learning Packages which use microchemistry activities to address general and inorganic chemistry. The Microchemistry Packages have been published by UNESCO in different languages, and have recently become available on the Internet for free access and use by UNESCO member states.

The organic chemistry materials open up new possibilities of using microscience kits at different educational levels to do practical laboratory work. In some countries, the educational curriculum allows for organic chemistry at secondary school level to be part of a general chemistry syllabus; in some other countries, organic chemistry is an independent part of chemical sciences at secondary level. Whatever the case may be, these educational materials will provide simple and cost-effective methods to do new practical laboratory work with the microscience kits.

We hope, too, that these Organic Chemistry Microscience Experiments will soon be installed on the Internet for free use.

Some years ago, Professor John Bradley, President of the International Foundation for Science Education (IFSE), Director of the RADMASTE Centre, and formerly Chairman of IUPAC-CTC, received the Pierre Crabbe IOCD Award for outstanding contributions to the advancement of science education in the developing countries. In his introductory statements which follow, you can find some concrete details on the new educational materials contained in this publication.

Finally, please let me express my hope that these new microscience materials will be successfully used in the different countries of the Globe, and that this will lead to different language versions being prepared so that all may have access to practical organic chemistry.

Professor Jan Marie Lehn
President, IOCD
Nobel Prize winner

INTRODUCTION

All over the world, science educators declare that practical experiences are an essential part of learning science. However, in many countries these experiences are not provided in the majority of their primary and secondary schools. There are several reasons for this: cost, safety, waste disposal and teacher preparation. In the last few years a global programme under the aegis of UNESCO, and in association with a number of organisations and donors, has attempted to address and overcome that situation. The programme has promoted the idea that practical experiences can be provided at much lower cost and with much lower environmental impact by working on a small scale and using less expensive materials.

The programme has had much success. When the idea has been introduced (so far in more than 70 countries) it has always had a positive reception. In several countries, local initiatives have taken the idea further through pilot projects and on to wider national implementation. This programme of introducing the concept is continuing.

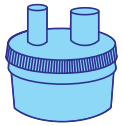
This satisfying record is however not enough. Not surprisingly, the original repertoire of experiences and low-cost equipment designs, has some limitations. In chemistry, for example, major areas of the subject have been ignored. One of these is organic chemistry. In several countries, the welcome accorded to the basic ideas, has been coupled with questions about these important missing areas. This workbook responds to these questions, by offering a limited repertoire of basic organic chemistry experiences that can be provided with low-cost small scale equipment. They have been developed at the RADMASTE Centre, University of the Witwatersrand in South Africa, the Centre which created the original materials referred to above. The worksheets and teacher guide layout will therefore be familiar to those who have used the previous chemistry books, which were published by UNESCO. Much of the equipment used will also be familiar, but there are some new and modified items.

In modern laboratories around the world, chemistry is increasingly done on the small scale. Organic chemistry has been one of the leaders in this trend and it is therefore fitting that this new publication offers experiences in this field to those who are starting their studies of it. We hope that both teachers and learners will enjoy the experiments, and will improve and modify them as a result of their own experience. Although many of the experiments can be carried out at the secondary school level, it is suggested that some of the activities be attempted by tertiary level students only.

Professor JD Bradley
Director, RADMASTE Centre &
UNESCO Associated Centre for
Microscience Experiments

COMPONENTS OF THE RADMASTE ORGANIC MICROCHEMISTRY KIT


microspatula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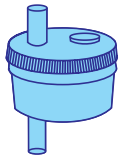
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silicone tube


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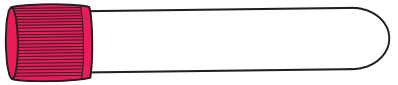
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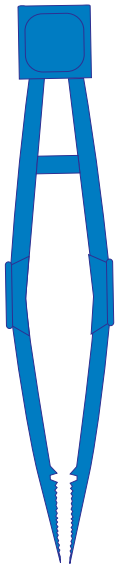
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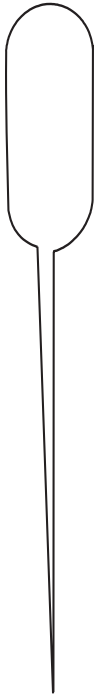
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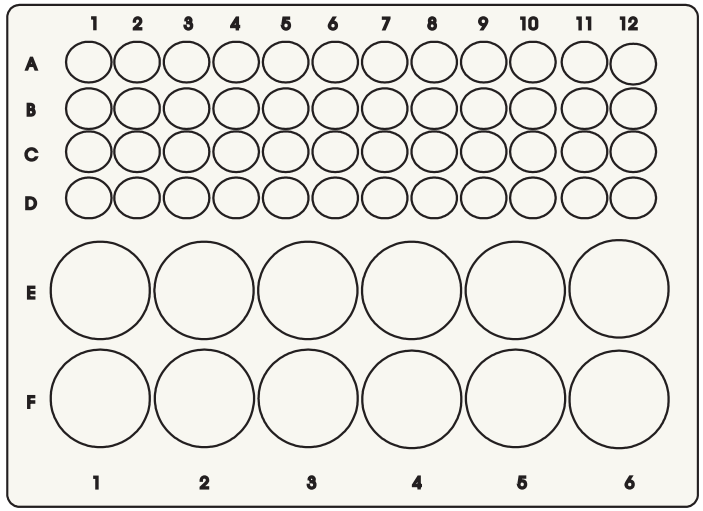

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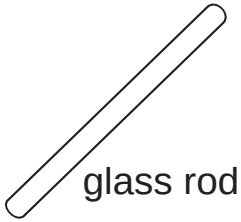
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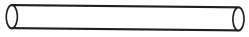
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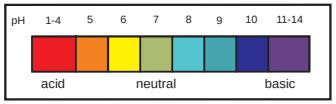

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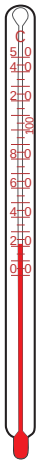
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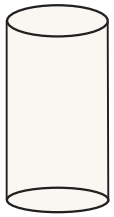
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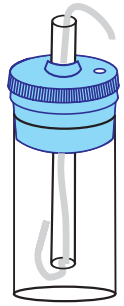
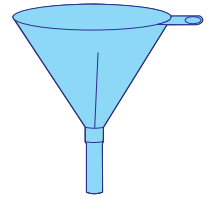

microburner

string


microfunnel

wire vial-holder

microstand

syringe

CHAPTER 1

Qualitative Testing of Organic Compounds

Qualitative Testing of Organic Compounds

PREPARING ESTERS

Esters are organic compounds formed by the reaction of an alcohol and a carboxylic acid (an organic acid) in the presence of a catalyst. The reaction can be represented by the general equation:

$$R_1OH + R_2COOH \rightarrow R_2COOR_1 + H_2O$$

where R_1 and R_2 refer to the hydrocarbon groups of the alcohol and acid respectively. The ester is formed with the elimination of water. Many esters occur naturally while others are produced by commercial synthesis. Esters have characteristic odours, a property which will be exploited in this experiment to identify specific esters. At the end of this exercise you should be able to recognise in which industries esters are used commercially.

REQUIREMENTS

Apparatus: 1 x Organic comboplate®; 6 x propettes; 1 x plastic microspatula; 1 x glass rod; 1 x microburner; Matches; Paper towelling; A pair of scissors (optional); Gloves (rubber or latex).

Chemicals: Ethanol ($C_2H_5OH(\ell)$); Methanol ($CH_3OH(\ell)$); 1-Pentanol (amyl alcohol - $C_5H_{11}OH(\ell)$); Isobutanol ($(CH_3)_2CHCH_2OH(\ell)$); Glacial acetic acid (ethanoic acid - $CH_3COOH(\ell)$); Salicylic acid ($C_6H_4OHCOOH(s)$); Formic acid ($HCOOH(\ell)$); Sulphuric acid ($H_2SO_4(aq)$) [18 M]; Tap water.

PROCEDURE

1. Cut or tear a piece of paper towel into smaller pieces. You will need about 12 small pieces of towelling.
2. Fill half of a clean propette with glacial acetic acid. Fill half of another propette with ethanol.

Always keep the stem of a propette containing an organic liquid facing downwards!

Hint: You may wish to label the propettes because all the chemicals used in the experiment are colourless.

3. Place one drop of the glacial acetic acid onto a small piece of paper towel. Cautiously hold the piece of towel close to your nose and smell the acid. *(See Question 1)*

Glacial acetic acid is a nose and eye irritant. Be very careful when smelling the acid!

4. Similarly, place a drop of the ethanol onto a different piece of paper towel and carefully smell the alcohol. *(See Question 1)*
5. Dispense 5 drops of the ethanol into well A1 of the organic comboplate®. Add 5 drops of glacial acetic acid to the ethanol in A1.
6. Use a clean propette to add 1 drop of concentrated (18 M) sulphuric acid to A1.
7. Light the microburner and place it on one side away from the comboplate®.

Most organic solvents are flammable! Ensure that all propettes and bottles containing organic substances are placed away from the flame of the microburner.

Wave one end of the glass rod five or six times through the flame of the microburner.

8. Carefully immerse the hot end of the glass rod into well A1. Do not boil the contents of the well! Stir the glass rod in the mixture in the well to distribute the heat throughout the mixture.
9. Dry the end of the rod. Repeat the immersion heating process another two times.

Note **As an alternative to immersion heating, a boiling water bath can be used. Pour boiling water into a plastic container (e.g. a 2 litre ice cream container) and carefully float the comboplate® in the water for approximately three minutes.**

10. Lift up the comboplate® and wave your hand across well A1 to smell the contents of the well. If you find it difficult to detect an odour in this way, suck up the contents of A1 with a clean propette and place a few drops onto a small piece of paper towel. Carefully inhale and smell the product. *(See Question 1)*
11. Use paper towel to absorb any liquid from well A1 and discard this into a waste container.

12. Use the table below to add the other carboxylic acids and alcohols in the proportions shown. Remember to smell the starting materials and resulting esters so that you can identify the odours of each.

Mixture/Well	Quantity of Alcohol	Quantity of Organic Acid	Quantity of H_2SO_4(aq)
Methanol and salicylic acid (Well A3)	5 drops	1 small spatula using narrow end of microspatula	1 drop
Pentanol and ethanoic acid (Well A5)	5 drops	5 drops	
Isobutanol and formic acid (Well A7)	5 drops	5 drops	

13. After adding the drop of concentrated H_2SO_4(aq) to each mixture, heat each mixture using the glass rod as described above.
14. Make sure that you dry out each well with paper towel after the odour of the ester has been identified, so that you do not become confused by the mixing of the odours of different esters.

Deposit all organic waste into a waste jar. Rinse the comboplate® and propettes with water. Extinguish the microburner!

QUESTIONS

Q1. Prepare a table like Table 1 in your workbooks. Complete the table by identifying the odours of the starting materials and products. You may compare the odour to that of a familiar product, or name the substance which has a similar odour to that of the acid, alcohol or ester. Name each of the esters formed during the reactions.

TABLE 1: DESCRIPTION OF THE ODOURS OF EACH ALCOHOL, CARBOXYLIC ACID AND THE RESULTING ESTER WHEN THESE ARE REACTED

ALCOHOL AND DESCRIPTION OF ODOUR	CARBOXYLIC ACID AND DESCRIPTION OF ODOUR	DESCRIPTION OF ODOUR OF PRODUCT	NAME OF ESTER
Ethanol	Glacial acetic acid/Ethanoic acid		
Methanol	Salicylic acid		
Pentanol/Amyl alcohol	Glacial acetic acid/Ethanoic acid		
Isobutanol/2 methyl propanol	Formic acid		

Q2. What is the name given to the type of reaction by which esters form from a carboxylic acid and an alcohol?

Q3. What evidence is there that esters formed in the reactions studied?

Q4. Complete the following predict, explore, explain exercise to determine the role of the sulphuric acid in each of the reactions:

PREDICT

Will an esterification reaction take place in the absence of concentrated sulphuric acid?

EXPLORE

1. Place 5 drops of ethanol into well A9, followed by 5 drops of glacial acetic acid.
2. Light the microburner and pass one end of the glass rod through the flame five or six times.
3. Heat the contents of A9 by immersion. Repeat the process three times.
   If a water bath is used, float the comboplate® in the boiling water for 3 minutes.
4. Smell the odour by either wafting the vapour from the well towards you, or by placing a drop of the mixture from A9 onto a piece of paper towel and smelling it carefully.

Has the expected ester been formed? How do you know this?

EXPLAIN

Was your prediction correct?
What is the role of concentrated sulphuric acid in the esterification reaction?

Q5. The introduction to this experiment shows the general equation for an esterification reaction. Use the equation as a guide to write down balanced equations representing each of the esterification reactions you have performed. The formulae of each alcohol and carboxylic acid are given under the chemical requirements. If possible, use a different colour pen to show the alcohol.

Q6. Using the description of the odours of each ester you have prepared in this experiment, choose two industries in which you think esters are used commercially. Give reasons for your answers.

Q7. If possible, obtain the containers of some food and cosmetic products. Read the lists of ingredients and identify any esters which appear on the labels.

Q8. Do all ethyl esters have the same smell? Do all formates have the same smell? Design, carry out and report on a piece of research to answer these questions.

FUNCTIONAL GROUP ANALYSIS

Tests for Aldehydes and Ketones

Part 1: The 2,4-Dinitrophenylhydrazine Test for Aldehydes and Ketones

REQUIREMENTS

Apparatus: Organic comboplate, 6 thin stemmed propettes, 6 plastic microspatulas, Gloves (rubber or latex).

Chemicals: 2,4-Dinitrophenylhydrazine (2,4-DNP) reagent (Brady's reagent), Methanol ($CH_3OH(\ell)$), Aqueous solution of formaldehyde ($CH_2O(aq)$), Propanone (acetone -$(CH_3)_2CO(\ell)$), Formic acid ($HCOOH(\ell)$), Ethyl acetate ($CH_3COOCH_3(\ell)$), Any other aliphatic aldehydes and/or ketones (optional).

Note *Any other aliphatic aldehyde or ketone may be tested with Brady's Reagent.*

PROCEDURE

Before beginning this experiment, you may find it helpful to label the propettes because most of the chemicals used are colourless.

1. Use a clean propette to add 8 drops of the 2,4-DNP reagent (Brady's reagent) to each of wells A1 to A6.
2. Add 2 drops of methanol to the reagent in well A1.

Always keep the stem of a propette containing an organic liquid facing downwards!

3. Observe the immediate reaction upon adding methanol to the well. Record your observations in Table 2. *(see Question 1)*
4. Stir the solution in A1 to thoroughly mix the contents if no precipitate has yet formed.
5. Add 2 drops of a solution of formaldehyde to well A2 using a clean propette. Observe the immediate reaction upon adding the formaldehyde and record the observations in Table 2 as before. Stir the solution in A1 with a clean microspatula if necessary.
6. Place 2 drops of each of the compounds listed below into wells A3 to A6 respectively (see Table 1). Remember to write down your immediate observations in Table 2. Stir each of the solutions with a clean microspatula.

Table 1

TEST SUBSTANCE	WELL
Propanone (acetone)	A3
Formic acid	A4
Ethyl acetate	A5
Any other aliphatic aldehyde or ketone (optional)	A6

7. Leave the comboplate to stand for about 5 to 6 minutes. Examine each well to determine whether a precipitate has formed during the 5 minute standing period. If no precipitate has formed in a well after such time, use a clean microspatula to stir the contents once more. Leave the solutions to stand for another minute before recording your results in the relevant column in Table 2 *(see Question 1)*.

Note **As soon as all results have been recorded, discard the contents of the wells into a waste container. If the precipitates are left in the comboplate7, they will cause staining which is difficult to remove. Rinse the comboplate thoroughly with water. If the precipitates adhere to the inside of the wells, flush the comboplate with boiling water. Stubborn stains can be removed by placing a few drops of glacial acetic acid into each well. Wrap a small strand of cotton wool around the pointed end of a wooden skewer and use it to rub away the stains in the wells containing the acetic acid. Alternatively, use a cotton wool bud.**

Acetic acid irritates the membranes of the nose and eyes. Be very careful when using this solvent.

FUNCTIONAL GROUP ANALYSIS

Tests for Aldehydes and Ketones

Part 1: The 2,4-Dinitrophenylhydrazine Test for Aldehydes and Ketones

QUESTIONS

Q1. What do you observe immediately and after 5 minutes? Prepare a table like the one below.

TABLE 2: TESTING OF VARIOUS COMPOUNDS WITH 2,4-DNP REAGENT

COMPOUND	IMMEDIATE REACTION WITH 2,4 DNP REAGENT	REACTION AFTER STANDING (~ 5 MINUTES)
Methanol		
Formaldehyde (aqueous)		
Propanone		
Formic acid		
Ethyl acetate		
Other aliphatic ketone or aldehyde (optional)		

Q2. Name the class to which each of the compounds used in this experiment belongs (eg. alcohol, alkane, ester, etc.)

methanol

formaldehyde

propanone

formic acid

ethyl acetate

other

Q3. Use the results in Table 2 and your answer to question 2 to explain how the 2,4-DNP test helps to distinguish aldehydes and ketones from other -C-O- and -C=O containing compounds.

FUNCTIONAL GROUP ANALYSIS

Tests for Aldehydes and Ketones

Part 2: Tests to Distinguish between Aldehydes and Ketones

2.1: Reduction of Ammoniacal Silver Nitrate Solution - Tollen's Test

REQUIREMENTS

Apparatus: Organic comboplate, 6 thin stemmed propettes, 4 plastic microspatulas, Gloves (rubber or latex).

Chemicals: 0.3 M silver nitrate solution ($AgNO_3(aq)$); 1 M sodium hydroxide solution (NaOH(aq)); 1 M ammonia solution ($NH_3(aq)$); Aqueous solution of formaldehyde ($CH_2O(aq)$); Propanone (acetone -$(CH_3)_2CO(l)$; Any other aliphatic aldehydes and/or ketones (optional), 2 M nitric acid ($HNO_3(aq)$).

Note *Any other aliphatic aldehyde or ketone may be tested using Tollen's reagent.*

PROCEDURE

1. Use a clean propette to fill one third of each of wells F1 and F2 with the 0.3 M silver nitrate solution.
2. Add 1 drop of 1 M sodium hydroxide solution to each well. Observe what happens.
3. Add approximately 20 drops of 1 M ammonia solution to well F1. Stir the contents of the well thoroughly with a clean microspatula to dissolve most of the brown precipitate. If the solid has not completely dissolved, add ammonia solution one drop at a time with stirring until the precipitate just dissolves. Do not add an excess of the ammonia solution! (25 drops maximum should be enough.)
4. Repeat step 3 with the solution in well F2.
5. Using a clean propette, place 1 to 3 drops of the aqueous formaldehyde into the solution in well F1. Stir gently with a microspatula.
6. Similarly, place 1 to 3 drops of the propanone into well F2 and stir gently with a clean microspatula. *(See Question 1)*
7. Allow the comboplate to stand for 5 to 10 minutes. After this time, hold the comboplate above your head and view wells F1 and F2 from under the comboplate. Tilt the comboplate towards the light to observe the walls of wells F1 and F2. Write down your observations.
8. Bring an object such as your finger or a microspatula close to the bottom of each well. What do you notice?

Note **Do not leave the precipitates to dry in the wells, as explosive compounds called silver fulminates may be formed! Rinse each well with a small volume of 2 M nitric acid, which dissolves the silver mirror and destroys the dangerous silver fulminates. Empty any solid into a waste container and rinse the comboplate thoroughly with water. If the walls of the wells are covered with a black residue, wrap a small piece of cotton wool around the pointed end of a wooden skewer and use it to rub the wells clean. Alternatively, use a cotton wool bud.**

FUNCTIONAL GROUP ANALYSIS

Tests for Aldehydes and Ketones

Part 2: Tests to Distinguish between Aldehydes and Ketones

2.1: Reduction of Ammoniacal Silver Nitrate Solution - Tollen's Test

QUESTIONS

Q1. What do you observe after 5 to 10 minutes? Prepare a table like the one below.

TABLE 3: TESTING OF ALDEHYDES AND KETONES WITH AMMONIACAL SILVER NITRATE SOLUTION

NAME OF ALDEHYDE/KETONE	REACTION WITH AMMONIACAL SILVER NITRATE SOLUTION
Formaldehyde (aqueous)	
Propanone	
*Butyraldehyde	

Q2. How can Tollen's test be used to differentiate between an aldehyde and a ketone?

Q3. The reaction which occurs when an aldehyde is added to Tollen's reagent is:

$$RCHO(\ell) + 2[Ag(NH_3)_2]OH(aq) \rightarrow RCOONH_4(aq) + 2Ag(s) + 3NH_3(aq) + H_2O(\ell)$$

i. What type of reaction is this?

ii. Explain your answer to i above.

FUNCTIONAL GROUP ANALYSIS

Tests for Aldehydes and Ketones

Part 2: Tests to Distinguish between Aldehydes and Ketones

2.2: Schiff's Test to Distinguish between Aldehydes and Ketones

REQUIREMENTS

Apparatus: Organic comboplate, 3 thin stemmed propettes, 2 plastic microspatulas, Gloves (rubber or latex)

Chemicals: Schiff's Reagent (fuchsin aldehyde reagent); Aqueous solution of formaldehyde ($CH_2O(aq)$); Propanone (acetone -$(CH_3)_2CO(\ell)$); Any other aliphatic aldehydes and/or ketones (optional).

Note *Fresh Schiff's reagent should be colourless. If the reagent is pale purple/pink in colour, it can still be used provided a colour comparison is made with the reagent in the experiment (ask your teacher for details). If the reagent is deep purple in colour, it is useless and should be discarded.*
Any other aliphatic aldehyde or ketone may be tested using Schiff's reagent.

PROCEDURE

1. Use a clean propette to add 10 drops of Schiff's reagent to each of wells A1 and A3. *(see Question 1)*

Note **Keep the bottle containing Schiff's reagent closed at all times, and do not expose the solution to heat and light.**

2. Place 1 drop of the formaldehyde solution into well A1 and stir the contents gently with a microspatula.
3. Place 1 drop of propanone into well A3 and stir gently with a clean microspatula.
4. Observe what happens in each well and record the results within two minutes.

Note **Any colour change that takes place after two minutes from the time of addition of the aldehyde/ketone to the Schiff's reagent, must be disregarded.** *(see Question 1)*

Note **As soon as the results have been recorded, discard waste into a waste container and rinse out the comboplate thoroughly with water. Do not leave the comboplate to stand without washing as the wells will become stained. Stubborn stains can be removed by adding a few drops of 5.5 M hydrochloric acid to the affected wells, and then rubbing the colour away with a wooden skewer which has cotton wool wrapped around its pointed end.**

FUNCTIONAL GROUP ANALYSIS

Tests for Aldehydes and Ketones

Part 2: Tests to Distinguish between Aldehydes and Ketones

2.2: Schiff's Test to Distinguish between Aldehydes and Ketones

QUESTIONS

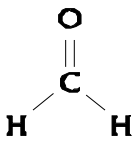
Q1. What do you observe in each well? Prepare a table like the one below.

TABLE 4: TESTING OF ALDEHYDES AND KETONES WITH SCHIFF'S REAGENT

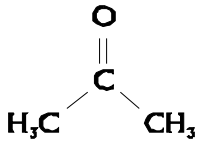
ALDEHYDE/KETONE TESTED	REACTION WITH SCHIFF'S REAGENT
Formaldehyde (aqueous)	
Propanone	
Other aliphatic aldehyde or ketone (optional)	

Q2. Based upon the results in Table 4 above, explain how the Schiff's test allows one to distinguish between aldehydes and ketones.

Q3. Examine the structures of formaldehyde and propanone below.



Formaldehyde

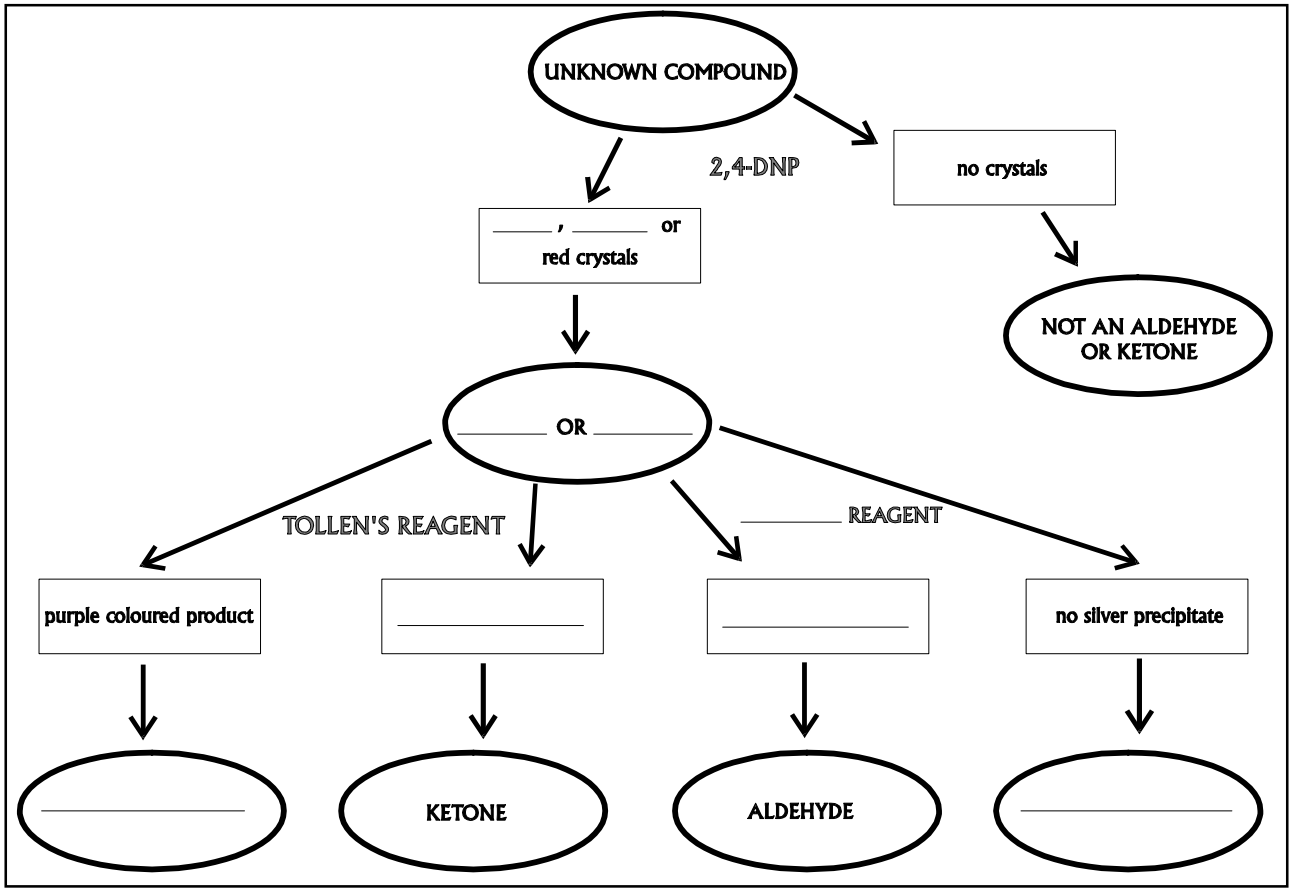


Propanone

ii. When formaldehyde reacts with Tollen's reagent, what is it converted to? Write equations to illustrate your answer.

ii. Explain why propanone cannot react in the same way.

Q4. The figure below shows a scheme for testing substances to check whether they are aldehydes or ketones. Complete the chart by filling in the missing words related to the experiments completed in this worksheet.



FUNCTIONAL GROUP ANALYSIS

The Iodoform Reaction

REQUIREMENTS

Apparatus: Organic comboplate, 6 thin stemmed propettes, 6 plastic microspatulas, Gloves (rubber or latex).

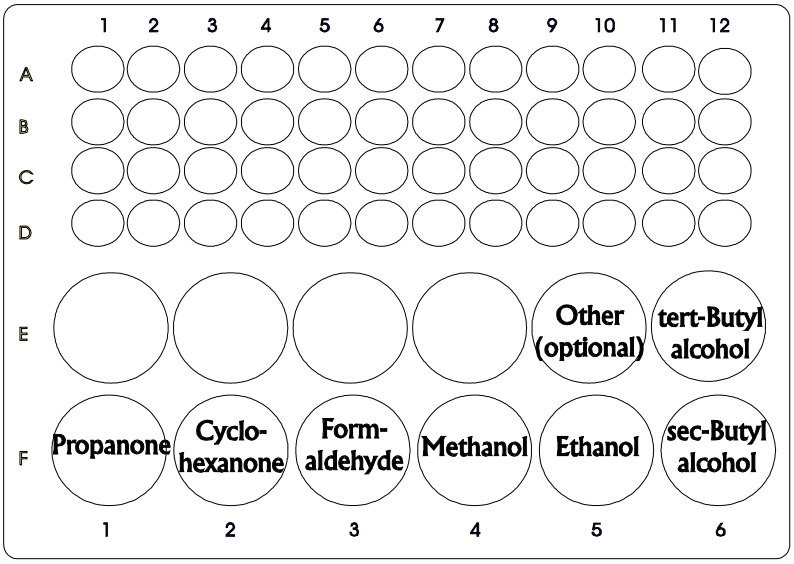
Chemicals: Potassium iodide-iodine reagent ($KI/I_2(aq)$), 1 M sodium hydroxide solution (NaOH(aq)), Propanone (acetone - $(CH_3)_2CO(\ell)$), Cyclohexanone ($C_5H_{10}CO(l)$), Aqueous solution of formaldehyde ($CH_2O(aq)$), Methanol ($CH_3OH(\ell)$), Ethanol ($C_2H_5OH(\ell)$), Secondary butyl alcohol ($CH_3CH(OH)CH_2CH_3(\ell)$), Tertiary butyl alcohol (2-methyl-2-propanol -$(CH_3)_3COH(\ell)$), Any other alcohol (primary, secondary or tertiary), or ketone (optional).

Note *Any other alcohol or ketone may be tested with the KI/I_2 and NaOH (hypoiodite) solutions.*

PROCEDURE

Before beginning this experiment, you may find it helpful to label the propettes because most of the chemicals used are colourless. It may be easier to perform this experiment in pairs as there are a number of compounds that require testing. Two students can then share the testing of the compounds.

1. Using the diagram below as a guide, place 5 drops of each of the organic compounds to be tested into the wells as shown. Remember to use a clean propette for each compound.



Always keep the stem of a propette containing an organic liquid facing downwards! Propanone, especially, tends to squirt easily from the propette stem so that drops of the compound will fall into the well without the need to apply pressure to the propette bulb.

2. Add 15 drops of 1 M NaOH(aq) to each well. Stir the contents of each well with a clean microspatula.
3. Add 25 drops of the potassium iodide-iodine reagent to the propanone in well F1. Stir the contents of the well as soon as the reagent has been added.
4. Repeat step 3 with all of the compounds to be tested. Remember to stir the contents of one well before adding the potassium iodide-iodine reagent to the following well.
5. Allow the comboplate to stand for about 2 to 3 minutes. After this time, note the appearance of the contents of each well and record the results in Table 1. *(See Question 1)*

6. Once the results have been recorded, wait for another 5 to 10 minutes. Lift the comboplate upwards and observe the wells from the underside of the comboplate. *(See Question 2)*

Note **If a precipitate cannot be seen clearly, try one or both of the following methods to show that there is a solid deposit in certain wells.**

7. Use clean microspatulas to scratch the bottom of those wells in which turbidity is present. Lift the comboplate up again and observe the wells from underneath. (*see Question 3a)*

8. Pour out the contents of the comboplate into a container for organic waste. Examine each of the wells. *(see Question 3b)*

9. Smell the contents of one of the wells where a precipitate is present. *(see Question 4)*

Note **As soon as all results have been recorded, discard the contents of the wells into a waste container. Rinse the comboplate thoroughly with water. If the precipitates adhere to the bottom of the wells, wrap a small strand of cotton wool around the pointed end of a wooden skewer and use it to remove solid residue. Alternatively, use a cotton wool bud or a small cleaning brush if available.**

FUNCTIONAL GROUP ANALYSIS

The Iodoform Reaction

QUESTIONS

Q1. What do you observe after 2-3 and after 5-10 minutes? Prepare a table like the one below.

TABLE 1: TESTING OF VARIOUS COMPOUNDS WITH HYPOIODITE (I_2/KI - NaOH) SOLUTION

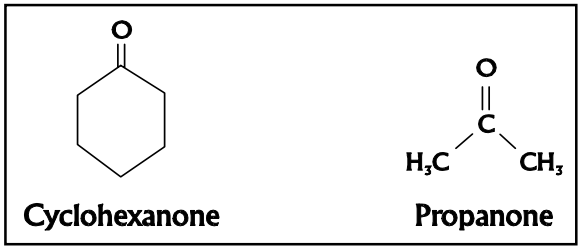
COMPOUND	APPEARANCE OF WELL CONTENTS AFTER 2 TO 3 MINUTES	PRECIPITATE (CHI_3(s)) FORMATION?
Propanone		
Cyclohexanone		
Formaldehyde (aqueous)		
Methanol		
Ethanol		
Secondary butyl alcohol		
Tertiary butyl alcohol		
Any other alcohol or ketone (optional)		

Q2. What do you notice in the wells where turbidity was observed earlier?

Q3a. What do you see?
Q3b. What do you notice?

Q4. Describe the odour.

Q5. Examine the structures of propanone and cyclohexanone along side. What is the difference in structure between these two ketones, besides the fact that one ketone is cyclic and the other aliphatic?



Q6. Based upon your answer to question 1 and the observations made in this experiment, how does the iodoform test help to distinguish methyl ketones from non-methyl ketones?

Q7. The reaction of a methyl ketone with iodine solution in the presence of aqueous sodium hydroxide is shown in the equation below:

$$RCOCH_3(\ell) + 3I_2(aq) + 4NaOH(aq) \rightarrow RCOONa(aq) + CHI_3(s) + 3NaI(aq) + 3H_2O(\ell)$$

(where R = H, alkyl or aryl group

i. Which part of the ketone molecule has been involved in the formation of an iodoform molecule?
ii. Use your answer to i to explain why cyclohexanone gives a negative reaction in the iodoform test.

Q8. Formaldehyde gives a negative result in the iodoform test. Do you think that the iodoform test could be used to distinguish methyl ketones from all aldehydes? Explain your answer with reference to the equation given in question 3.

Q9. The equation for the initial reaction between an alcohol (primary or secondary) and iodine solution in the presence of aqueous sodium hydroxide is:

$$\mathbf{RCH(OH)R'(\ell) + I_2(aq) + 2NaOH(aq) \rightarrow RCOR'(aq) + 2NaI(aq) + 2H_2O(\ell)}$$

where R and R' = H, alkyl or aryl group

i. According to the equation, which alcohols should react with the hypoiodite solution?

ii. Use the equation to explain why certain alcohols are able to give a positive iodoform reaction.

iii. Why does ethanol give a positive iodoform test but methanol does not? *(Hint: look at the reaction equation)*

Q10. Refer back to Table 1. Write down the reactions taking place for those ketones and alcohols which gave a positive iodoform test.

Q11. You are working in a company laboratory. You have been asked to use 1-propanol in a particular experiment. You find a cupboard in which there is a shelf for alcohols, but the bottles are not labelled. You know that ethanol and 1-propanol are used often. Explain how you will use the iodoform test to locate a bottle of 1-propanol in the presence of bottles of ethanol. Predict the outcome of your investigation and refer to the structures of the alcohol compounds when necessary.

FUNCTIONAL GROUP ANALYSIS

Tests for Identifying and Classifying Alcohols

Part 1: The Sodium Test: Using Metallic Sodium to Distinguish Alcohols from Ethers

REQUIREMENTS

Apparatus: Organic comboplate, 4 long stemmed propettes, 1 plastic microspatula, 1 x pair of plastic forceps; 1 x sharp blade or knife ; Gloves (rubber or latex).

Chemicals: Ethanol ($C_2H_5OH(\ell)$), Diethyl ether ($(C_2H_5)_2O(\ell)$), Anhydrous magnesium sulphate ($MgSO_4(s)$), Sodium metal (Na(s)) stored under liquid paraffin.

Note *The sharp blade or knife is required to cut the sodium into smaller pieces, and to remove the oxide layer from the metal.*

PROCEDURE

Water interferes with this experiment! Ensure that the comboplate is thoroughly dry. Remove all traces of water from the large wells using paper towel. The four propettes required for dispensing the alcohol and ether must also be dry. It is a good idea to label the propettes to be used for the alcohol and ether as they are both colourless liquids.

1. Fill approximately 2 of well F1 with ethanol from a clean, *dry* propette.
2. Fill 2 of well F2 with diethyl ether using another clean, *dry* propette.

CAUTION! Always keep the stem of a propette containing an organic liquid facing downwards! Diethyl ether, especially, squirts easily from the propette stem without the need to apply pressure to the propette bulb.

3. Use the spoon end of a microspatula to add 10 spatulas of anhydrous magnesium sulphate to each of wells F1 and F2.
4. Remove a small piece of sodium from the liquid paraffin with the plastic forceps (tweezers) and place it on some paper towel.
5. Use a sharp blade or knife to cut the sodium into smaller pieces so that each piece is about the size of half a grain of rice (2 mm x 2 mm x 2 mm).
6. If there is a white layer of oxide surrounding the outer edges of the metal pieces, scrape this away using the blade or knife.
7. Select another clean, *dry* propette and use it to remove most of the ethanol from well F1, being careful not to suck up any of the magnesium sulphate at the bottom of the well. Place the ethanol into well F4.
8. Repeat step 6 with the diethyl ether in F2, using another *dry* propette. Transfer the ether to well F5. *(See Question 1)*
9. Use the plastic forceps to add a piece of sodium to the ethanol in F4. Similarly, add a piece of sodium to the ether in F5. The pieces of sodium must be similar in size for a proper comparison to be made between the ethanol and ether.
10. Observe what is happening in each well. Record the results after 5 to 10 minutes.

Note **As soon as all results have been recorded, destroy any unreacted sodium by adding a little ethanol to the well/s containing the metal. Rinse the comboplate thoroughly with water.**

FUNCTIONAL GROUP ANALYSIS

Tests for Identifying and Classifying Alcohols

Part 1: The Sodium Test: Using Metallic Sodium to Distinguish Alcohols from Ethers

QUESTIONS

Q1. Prepare a table like the one below and record your observations.

TABLE 1: TESTING OF ALCOHOL AND ETHER WITH METALLIC SODIUM

COMPOUND	OBSERVATIONS
Ethanol	
Diethyl Ether	

Q2. How can one distinguish between an alcohol and an ether using the sodium test?

Q3. Write down the equation for the reaction of ethanol with sodium.

Q4. Write down the reaction of water with sodium metal.

Q5. The reaction of anhydrous magnesium sulphate with water is as follows:

$$MgSO_4(s) + 7H_2O(\ell) \rightarrow MgSO_4.7H_2O(s)$$

i. What is the function of the anhydrous magnesium sulphate in the sodium test?

ii. Why is it important to add anhydrous magnesium sulphate to the alcohol and ether prior to reaction with sodium metal? (Hint: look at your answer to question 1 above.)

iii. Ether does not react with water. Explain why you observed a reaction when sodium was added to the diethyl ether. (Hint: look at your answer to question 1 above.)

iv. Use your answer to iii. to explain why there was a difference in the rates of reaction when sodium was added to ethanol and diethyl ether.

v. What is the name of the precipitate that settled at the bottom of the well containing the ether?

FUNCTIONAL GROUP ANALYSIS

Tests for Identifying and Classifying Alcohols

Part 2: Tests for Classifying Alcohols as Primary, Secondary and Tertiary

2.1 Lucas' Test for Differentiating Between Primary, Secondary and Tertiary Alcohols

REQUIREMENTS

Apparatus: Organic comboplate, 4 long stemmed propettes, 3 plastic microspatulas, 1 x water bath at 27 - 28^{0}C; 1 x thermometer; Gloves (rubber or latex).

Chemicals: *n*-butyl alcohol ($C_4H_9OH(\ell)$), Secondary butyl alcohol ($CH_3CH(OH)CH_2CH_3(\ell)$), Tertiary butyl alcohol (2-methyl-2-propanol - $(CH_3)_3COH(\ell)$), Lucas' Reagent (hydrochloric acid-zinc chloride reagent), Any other aliphatic or cycloaliphatic alcohol - primary, secondary or tertiary (optional), Hydrochloric acid ($HC\ell(aq)$, 11 M).

Note *A water bath is required since the desired reaction occurs at 27^{0}C to 28^{0}C. A simple water bath is best constructed by using a plastic container (such as a 2 litre ice cream container) into which a small volume of water is placed. The temperature of the water is regulated by mixing hot and cold water together. A thermometer should be used to ensure the water temperature is maintained at 27^{0}C to 28^{0}C.*
Any other aliphatic or cycloaliphatic alcohol may be tested with Lucas' Reagent.

PROCEDURE

If possible, label the propettes used to dispense the alcohols as they are all colourless and may be easily confused.

1. Set up a water bath as described in the Note above. Ensure that the water temperature remains at 27^{0}C to 28^{0}C.
2. Place 2 drops of *n*-butyl alcohol into well A1 using a clean propette.
3. Similarly, add 2 drops of secondary butyl alcohol to well A3 and 2 drops of tertiary butyl alcohol to well A5. Use a clean propette for each alcohol. *(See Question 1)*
4. Add 12 drops of Lucas' Reagent to A1 and stir the contents with a clean microspatula. Record any immediate reaction between *n*-butyl alcohol and Lucas' Reagent.
5. Repeat step 3 for the secondary butyl alcohol in well A3.
6. Repeat step 3 for the tertiary butyl alcohol in well A5.
7. Float the comboplate in the water bath at 27 to 28^{0}C for about 5 minutes. (**Do not push the comboplate into the water as the wells will be flooded.**)
8. After 5 minutes, remove the comboplate from the water bath and dry the outside with paper towel.
9. Observe each of the wells from above and from the underside of the comboplate to check for signs of turbidity. (Hold the comboplate towards the light if the plastic of the plate is obscuring any observations.) *(See Question 1)*
10. Place 2 drops of secondary butyl alcohol into well A7.
11. Place 2 drops of tertiary butyl alcohol into well A8.
12. Use a clean propette to add 8 drops of 11 M hydrochloric acid to each of wells A7 and A8.
13. Stir the contents of each well with a clean microspatula. *(see Question 2)*

14. Observe each well from above and from the underside of the comboplate. Hold the comboplate up to the light if necessary. Record your results.

Note **As soon as all results have been recorded, empty the contents of the wells onto a thick pile of paper towel. The organic solvents will evaporate and the paper towel can then be discarded. Alternatively pour the contents of the wells into a separate waste container and not into a container for general waste, because chlorinated organic waste should not be mixed with non-chlorinated waste. Rinse the comboplate thoroughly with water.**

FUNCTIONAL GROUP ANALYSIS

Tests for Identifying and Classifying Alcohols

Part 2: Tests for Classifying Alcohols as Primary, Secondary and Tertiary

2.1 Lucas' Test for Differentiating Between Primary, Secondary and Tertiary Alcohols

QUESTIONS

Q1. Prepare a table like the one below and record your results.

TABLE 2: REACTION OF PRIMARY, SECONDARY AND TERTIARY ALCOHOLS WITH LUCAS' REAGENT

ALCOHOL	IMMEDIATE REACTION WITH LUCAS' REAGENT	OBSERVATIONS AFTER 5 MINUTES AT 27 TO 28^{0}C
n-butyl alcohol		
secondary butyl alcohol		
tertiary butyl alcohol		
*other (optional)		

Q2. Prepare a table like the one below and record your observations.

TABLE 3: REACTION OF SECONDARY AND TERTIARY ALCOHOLS WITH CONCENTRATED HYDROCHLORIC ACID

ALCOHOL	OBSERVATIONS
secondary butyl alcohol	
tertiary butyl alcohol	

Q3. Classify the alcohols used in this experiment as primary, secondary or tertiary.

Q4. Use your answer to question 1 and the results in Table 2 to explain how mixing an alcohol of unknown structure with Lucas' Reagent can help one to determine whether it is primary, secondary or tertiary.

Q5. Write down an equation for the reaction of tertiary butyl alcohol with concentrated hydrochloric acid.

Q6. Explain why the solution became milky white in appearance after adding 11 M HCℓ(aq) to the tertiary butyl alcohol.

Q7. A student is given two alcohols with which to perform Lucas' Test. These are cyclohexanol and 2-methyl-2-propanol. The bottles containing each alcohol are labelled, but the propettes used are not. The student accidentally muddles the propettes. When drops of each alcohol are mixed with Lucas' Reagent and kept at 27^0 C, both solutions become turbid. How can the student find out which alcohol has been placed in which propette?

FUNCTIONAL GROUP ANALYSIS

Tests for Identifying and Classifying Alcohols

Part 2: Tests for Classifying Alcohols as Primary, Secondary and Tertiary

2.2 The Chromic Anhydride Test: Distinguishing Tertiary Alcohols from Primary and Secondary Alcohols Using Chromic Anhydride Reagent (Jones' Reagent)

REQUIREMENTS

Apparatus: Organic comboplate, 5 long stemmed propettes, 3 plastic microspatulas, Gloves (rubber or latex).

Chemicals: Pure acetone (propanone - $(CH_3)_2CO(l)$, *n*-butyl alcohol ($C_4H_9OH(l)$), Secondary butyl alcohol ($CH_3CH(OH)CH_2CH_3(l)$), Tertiary butyl alcohol (2-methyl-2-propanol - $(CH_3)_3COH(l)$), Chromic Anhydride/Chromium Trioxide Reagent, (Jones' Reagent), Any other alcohol - primary, secondary or tertiary (optional)

Note *The Chromic Anhydride Reagent, also called Jones' Reagent, is a solution of chromic anhydride (chromium trioxide - CrO_3) in dilute sulphuric acid.*
Any other alcohol may be tested with the Chromic Anhydride Reagent.

PROCEDURE

1. Place 5 drops of pure acetone into each of wells A1, A3 and A5.
2. Add 1 drop of n-butyl alcohol to well A1 and stir the contents with a clean microspatula.
3. Add 1 drop of secondary butyl alcohol to well A3 and stir with a clean microspatula. Do the same with the tertiary butyl alcohol in well A5. *(See Question 1)*
4. Add 1 drop of the Chromic Anhydride Reagent to the *n*-butyl alcohol in A1. Observe the well and record any reaction which occurs within 3 to 5 seconds of adding the reagent to the well.
5. Add 1 drop of the reagent to A3. Observe and record any reactions which may occur within 3 to 5 seconds.
6. Repeat the process for tertiary butyl alcohol in A5 and record any reactions occurring within 3 to 5 seconds of adding the reagent.

Note **Do not add the Chromic Anhydride Reagent to all the wells simultaneously as any reactions occurring within the 3 to 5 second time period may not be properly seen. Concentrate on one alcohol at a time. Disregard any reactions which occur after 5 seconds.**

7. As stated above, any reactions which occur after 5 seconds should be disregarded. However, note any ***changes in appearance*** of the well contents after the initial reaction has occurred. (Any changes should be recorded no longer than about 20 to 30 seconds after the initial reaction has taken place.)

Note **As soon as all results have been recorded, empty the contents of the wells onto a thick pile of paper towel. The organic solvents will evaporate and the paper towel can then be discarded. Alternatively pour the contents of the wells into a waste container. Rinse the comboplate thoroughly with water.**

FUNCTIONAL GROUP ANALYSIS

Tests for Identifying and Classifying Alcohols

Part 2: Tests for Classifying Alcohols as Primary, Secondary and Tertiary

2.2 The Chromic Anhydride Test: Distinguishing Tertiary Alcohols from Primary and Secondary Alcohols Using Chromic Anhydride Reagent (Jones' Reagent)

QUESTIONS

Q1. Prepare a table like the one below and record your observations.

TABLE 4: REACTION OF PRIMARY, SECONDARY AND TERTIARY ALCOHOLS WITH CHROMIC ANHYDRIDE REAGENT

ALCOHOL	REACTION WITHIN 3 TO 5 SECONDS OF ADDING CHROMIC ANHYDRIDE REAGENT	CHANGE IN APPEARANCE OF WELL CONTENTS AFTER INITIAL REACTION
n-butyl alcohol		
secondary butyl alcohol		
tertiary butyl alcohol		
*other (optional)		

Q2. How does the Chromic Anhydride Test distinguish primary and secondary alcohols from tertiary alcohols?

Q3i. Write down an equation for the reaction of *n*-butyl alcohol with Chromic Anhydride Reagent. (The reagent = $CrO_3 + H_2SO_4$)

Q3ii. What type of compound is formed in the reaction between a primary alcohol and Chromic Anhydride Reagent? (Hint: use the answer to 3i. above.)

Q4i. Write down an equation for the reaction of secondary butyl alcohol with Chromic Anhydride Reagent.

Q4ii. What type of compound is formed in the reaction between a secondary alcohol and the Chromic Anhydride Reagent? (Hint: use the answer to 4i. above.)

Q5. During the mixing of a primary or secondary alcohol with Chromic Anhydride Reagent, the following change occurs (equation unbalanced):

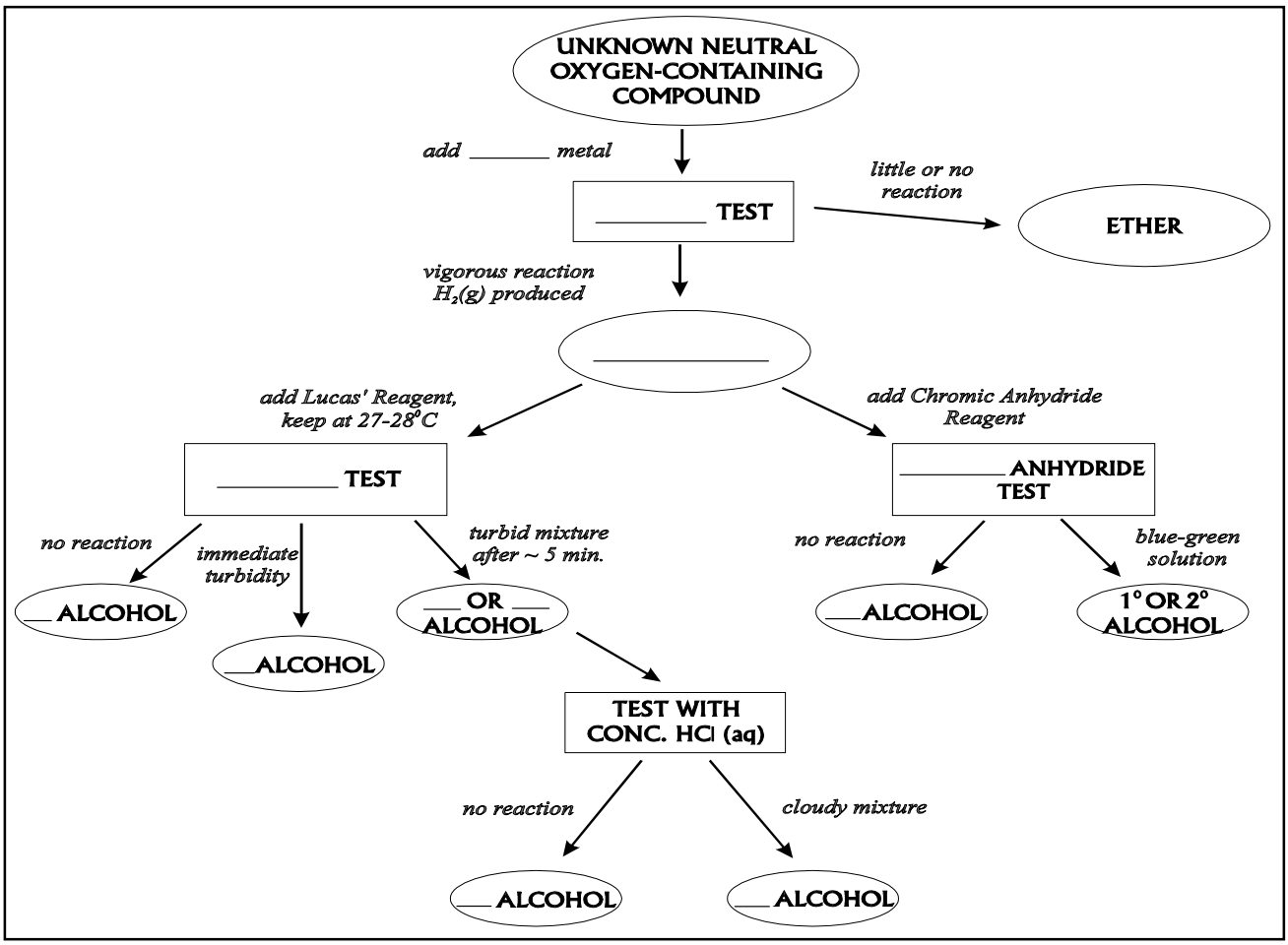
$$\mathbf{CrO_3(aq) + H_2SO_4(aq) \rightarrow Cr_2(SO_4)_3(s)}$$

Q5i. What kind of reaction has taken place? Explain by examining the partial reaction equation given.

Q5ii. What is the blue-green precipitate that was observed at the bottom of each well in which a reaction occurred?

Q5iii. Why did the test solutions containing primary and secondary alcohols, initially become blue-green in colour?

Q6. Draw the chart below and complete it by filling in the missing words related to the experiments carried out in this worksheet.



FUNCTIONAL GROUP ANALYSIS

Tests for Unsaturated Hydrocarbon Compounds

Part 1: The Bromine Test for Unsaturated Hydrocarbon Compounds

REQUIREMENTS

Apparatus: 1 x Organic comboplate®; 4 x propettes; 4 x plastic microspatulas; Gloves (rubber or latex).
Chemicals: Cyclohexane ($C_6H_{12}(\ell)$); Cyclohexene ($C_6H_{10}(\ell)$); Solution of bromine in water ($Br_2(aq)$); Any other alkane or alkene (optional).

PROCEDURE

Note **If the compounds to be tested are cyclohexane and cyclohexene, then an ordinary comboplate® may be used for this experiment because these compounds do not destroy the plastic of the comboplate®. If other alkanes and alkenes are tested, it is better to use the Organic comboplate® as the unknown compounds are likely to damage an ordinary comboplate®.**

This experiment should not be performed in an area with bright lighting as the bromine solution is decomposed by light, and light also interferes with the reactions being investigated.

Students should share propettes of the bromine solution. The bottle containing the bromine solution should be kept sealed at all times, and placed back in the cupboard or storage box so that it remains in the dark.

1. Use a clean propette to add 5 drops of cyclohexane to well A1.
2. Use another propette to add 5 drops of cyclohexene to well A2.

Always keep the stem of a propette containing an organic liquid facing downwards!

3. Add 5 drops of the bromine solution to each of wells A1 and A2. Stir the contents of each well vigorously using clean microspatulas. *(See Questions 1 and 2)*
4. Leave the comboplate® to stand for approximately 1 to 3 minutes. Observe wells A1 and A2 from the side of the comboplate® after this time. *(See Question 3)*
5. If time allows, leave the contents of wells A1 and A2 in the comboplate® until after Part 2 has been completed. Note any changes in the appearance of the mixtures and answer the questions given after Part 2.

If you are not going to continue with Part 2, discard the contents of wells A1 and A2 into a waste container. Rinse the comboplate® thoroughly with clean water.

FUNCTIONAL GROUP ANALYSIS

Tests for Unsaturated Hydrocarbon Compounds

Part 1: The Bromine Test for Unsaturated Hydrocarbon Compounds

QUESTIONS

Q1. What happens when the bromine solution is first added to each compound?

Q2. What do you notice when the bromine solution is stirred with each compound?

Q3. Prepare a table like Table 1 below. Record your observations for wells A1 and A2.

TABLE 1: TESTING OF SATURATED AND UNSATURATED COMPOUNDS WITH BROMINE SOLUTION

COMPOUND	APPEARANCE OF WELL CONTENTS AFTER 1 MINUTE	REACTION WITH BROMINE (Br_2 (aq))?
Cyclohexane		
Cyclohexene		

Q4. Write down the structure of cyclohexane. Is the compound saturated or unsaturated? Explain your answer.

Q5. Write down the structure of cyclohexene. Explain whether the compound is saturated or unsaturated.

Q6. Based on your answers to questions 4 and 5 above and on the results of the bromine test in your table, describe how mixing a bromine solution with a hydrocarbon compound of unknown structure can help one to tell whether it is saturated or unsaturated.

Q7. Write down a balanced equation for the reaction of bromine with cyclohexene. Is the product of the reaction saturated or unsaturated? Explain your answer by describing what type of reaction is involved.

Q8. Explain why the brown colour of bromine disappears when it is mixed with cyclohexene. (Hint: examine the equation written for question 7 above.)

Q9. The bromine addition test does not distinguish between a double or triple bond. The bromine always adds across the multiple bond to form a trans addition product/s. Write down the equation/s for the reaction/s involved when bromine is mixed with ethyne (acetylene).

FUNCTIONAL GROUP ANALYSIS

Tests for Unsaturated Hydrocarbon Compounds

Part 2: Baeyer's Test for Unsaturation: Reaction of Cold, Dilute Potassium Permanganate solution with Unsaturated Hydrocarbon Compounds

REQUIREMENTS

Apparatus: 1 x Organic comboplate®; 3 x propettes; 3 x plastic microspatulas; Gloves (rubber or latex).

Chemicals: Cyclohexane ($C_6H_{12}(\ell)$); Cyclohexene ($C_6H_{10}(\ell)$); Potassium permanganate solution ($KMnO_4(aq)$) [0.1 %]; Any other alkane or alkene (optional).

PROCEDURE

Potassium permanganate solutions are decomposed by heat and light. Students should share propettes of the permanganate solution, taking care to keep it cold. The bottle containing the $KMnO_4(aq)$ should be kept sealed at all times, and placed back in the cupboard or storage box so that it remains in a cool, dark environment.

1. Add 3 drops of cyclohexane to well A4.
2. Add 3 drops of cyclohexene to well A5.
3. Use a clean propette to add 6 drops of cold, 0.1% potassium permanganate solution to each of wells A4 and A5.
4. Immediately observe the contents of each well from the side of the comboplate®. *(See Question 1)*
5. Stir the contents of each well vigorously using clean microspatulas.
6. Raise the comboplate®. Note the appearance of the contents of each well to see if a colour change has taken place. *(See Questions 2 and 3)*

After completing Question 5, discard the contents of wells A1, A2, A4 and A5 into a waste container. Rinse the comboplate® thoroughly with clean water.

FUNCTIONAL GROUP ANALYSIS

Tests for Unsaturated Hydrocarbon Compounds

Part 2: Baeyer's Test for Unsaturation

QUESTIONS

Q1. What do you notice in each well immediately after adding the potassium permanganate solution?

Q2. Describe the appearance of the cyclohexane-permanganate mixture in well A4 after stirring.

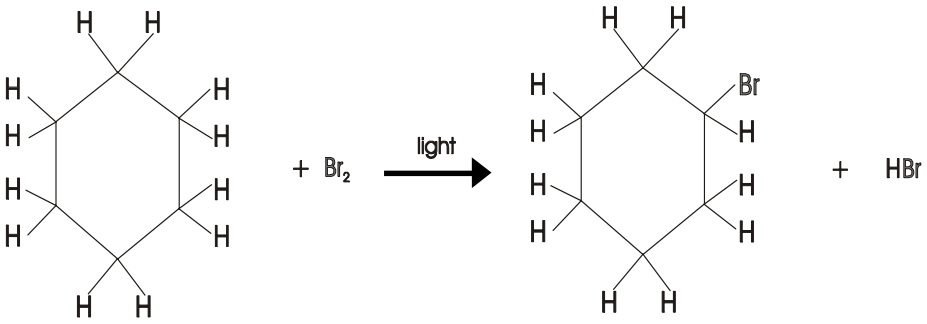
Q3. What has happened to the cyclohexene-permanganate mixture after stirring?

Q4. How can a dilute potassium permanganate solution help the chemist to distinguish between saturated and unsaturated hydrocarbon compounds?

Q5. If you have been able to leave the bromine-cyclohexane and bromine-cyclohexene mixtures from Part 1 in the comboplate® until the end of the lesson, answer the questions which follow:

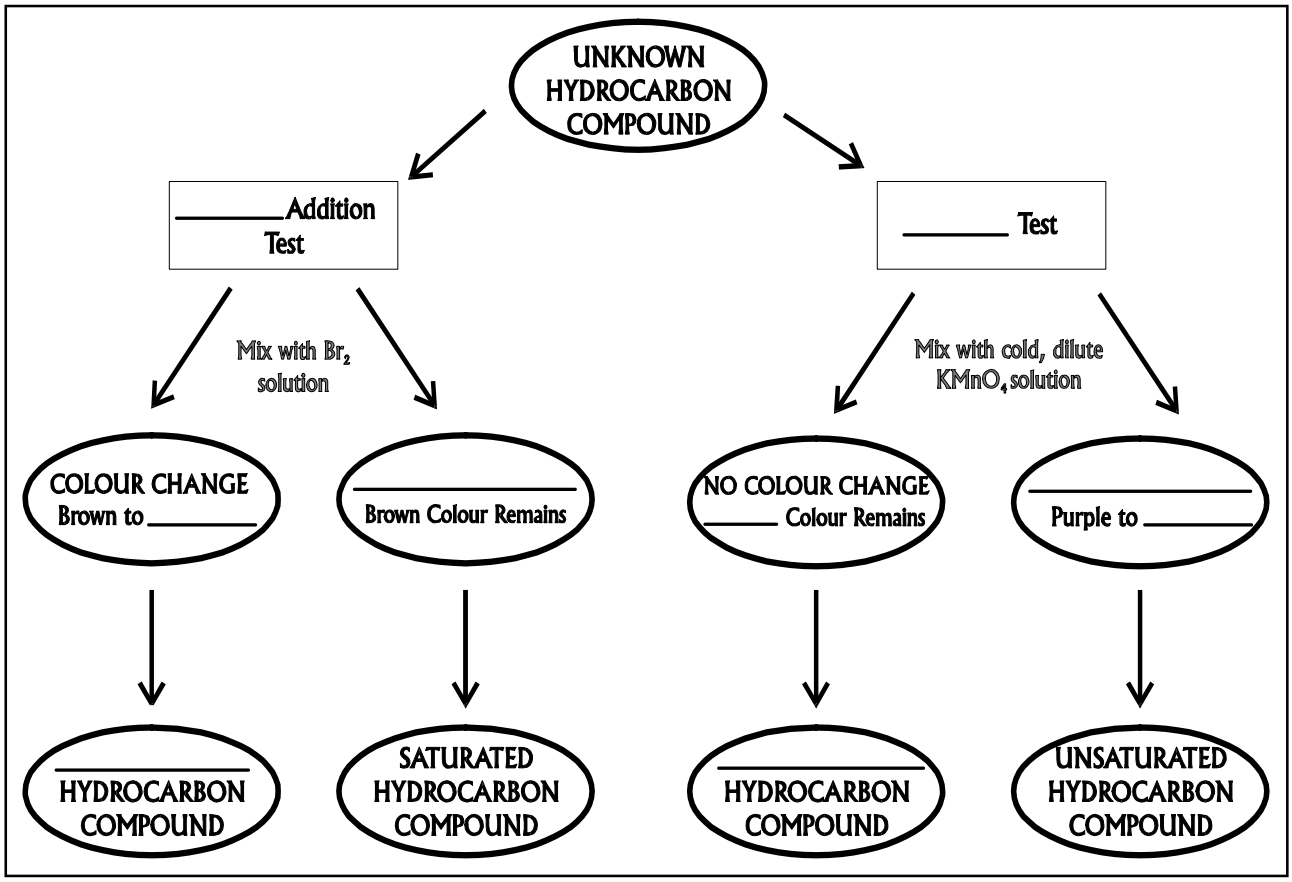
i. What do you notice about the appearance of the bromine-cyclohexane mixture?

ii. If cyclohexane is a saturated hydrocarbon compound, why has the brown colour faded? Use the following reaction equation to explain your answer.



iii. Name the kind of reaction shown in the given equation.

Q6. The figure below shows a scheme for testing hydrocarbon compounds to determine whether they are saturated or unsaturated. Draw the scheme and complete it by filling in the missing words related to the experiments completed in the worksheets of Parts 1 and 2.



Note: that even if the results of both the bromine and Baeyer's tests are positive, there remains an element of doubt.

INVESTIGATING INTERMOLECULAR FORCES IN ORGANIC COMPOUNDS BY MEASURING THE BOILING POINT

REQUIREMENTS

Apparatus: 1x Organic comboplate®; lid 1, 1 x glass vial, wire vial-holder, 1x microburner, matches, 1x paper clip, 2x elastic bands, 2x capillary tubes, 1 x 200°C thermometer, 1 x microretort stand, 1 x fusion tube, 1 x propette.

Chemicals: Boiling chips, paraffin oil, methylated spirits, ethanol ($C_2H_60(\ell)$), 1-butanol ($C_4H_{10}0(\ell)$), matches.

PROCEDURE

1) Determine the boiling point of 1-butanol (see General Procedures).

2) Determine the boiling point of ethanol (see General Procedures) *(See Questions 1-9)*

QUESTIONS

Q1. Report your boiling point temperatures for ethanol and 1-butanol.

Q2. Why is it important that the thermometer and fusion tube do not touch the bottom of the glass vial?

Q3. Why do bubbles emerge from the capillary tube? Explain why the boiling point is recorded when they stop.

Q4. Why is it necessary to record the atmospheric pressure?

Q5. Draw out the line drawings for 1-butanol and ethanol molecules.

Q6. What kinds of intermolecular forces occur between the 1-butanol molecules and between the ethanol molecules?

Q7. Explain the difference in boiling points between 1-butanol and ethanol in terms of the intermolecular forces.

EXTENSION QUESTION

Q8. Predict the boiling point of 1-propanol, giving an explanation of your prediction.

Q9. Predict the boiling point of 2-methyl-2-propanol, giving an explanation of your prediction.

MELTING POINT DETERMINATION

REQUIREMENTS

Apparatus: Organic comboplate, lid 1, glass vial, wire vial-holder, 1 x microburner, 2 x elastic bands, 2 x capillary tubes, 3 x microspatulas, syringe.

Chemicals: Boiling chips, paraffin oil, benzoic acid ($C_7H_6O_2$(s)), methylated spirits, matches.

PROCEDURE

1. Determine the melting point of benzoic acid (see General Procedures, Chapter 2).

QUESTIONS

Q1. Look up the melting point of benzoic acid in the literature. Compare it to your melting point and explain any differences.

Q2. Explain why the amount of sample in the capillary tube should be kept as small as possible for a melting point determination.

Q3. Draw out the line drawing for benzoic acid.

Q4. What kinds of intermolecular bonds occur between the benzoic acid molecules?

Q5. Do you think that the melting point of benzene will be higher or lower than that of benzoic acid ? Explain your reasonings.

CHAPTER 2

General Procedures

General Procedures

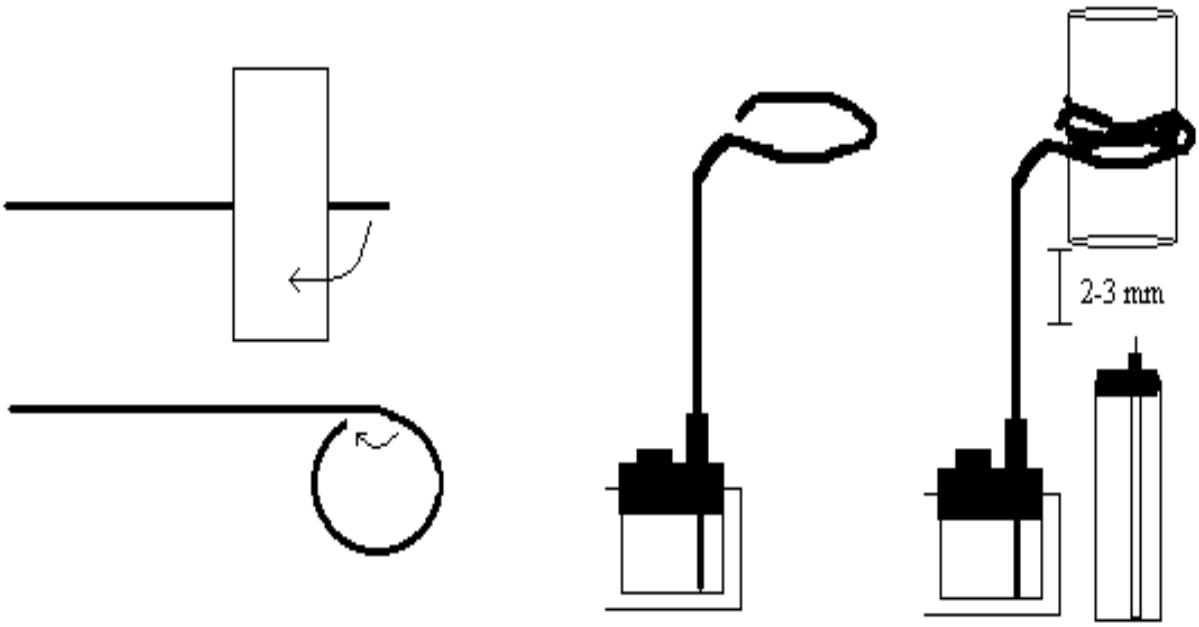
GENERAL PROCEDURES

The general procedures are experimental procedures or techniques that can be used in many, many experiments (especially organic chemistry ones) using the RADMASTE microchemistry kits.

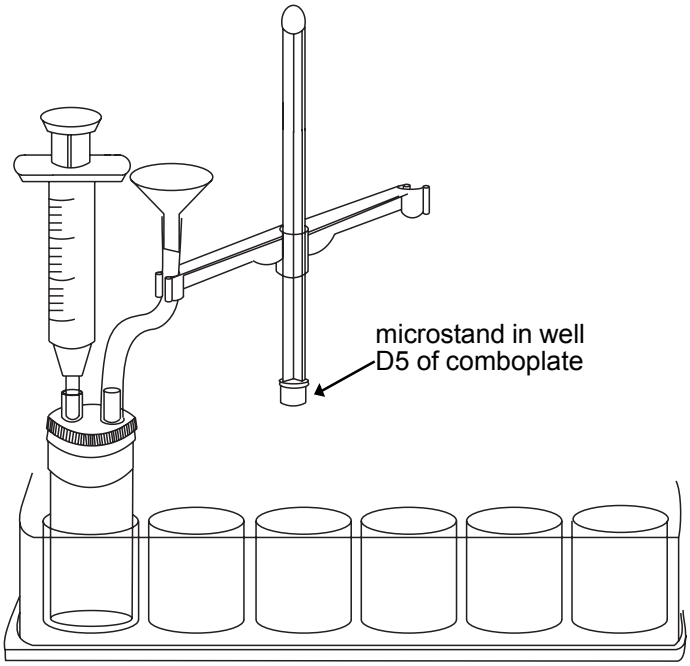
1. Assembling the Wire Microvial-holder

Apparatus: Organic comboplate, lid 1, glass vial, elastic band, microburner, iron wire (15 cm x 1.6 cm diameter).

1. Curl the 15 mm long piece of iron wire around the center of the glass vial, so that it forms a loop that fits exactly around the outside of the vial.
2. Place lid 1 into well E1.
3. Place the other end of the wire in the small opening in lid 1 so that it is touching the bottom of the comboplate as shown in the figure, and bend it at the end of the loop so that it can hold the glass vial vertically.
4. Wrap a piece of elastic around the centre of the glass vial and place it in the loop of the wire vial-holder.
5. Place the microburner under the vial.
6. Make sure the wire is bent in such a way so that the vial is about 2-3 mm above the flame of the microburner.



2. Vacuum Filtration and Washing of the Collected Solid



Apparatus: Organic comboplate, lid 1, glass vial with solid-liquid mixture to be filtered, silicone tubing, plastic microburner vial, cotton wool (or string), microfunnel, paper clip, syringe, glass rod, plastic microretort stand.

1. Unfold a paper clip.
2. Place a small piece of cotton wool into the opening of the microfunnel.
3. Force the cotton wool into the stem of the microfunnel using an unfolded paper clip. (In some experiments, string is used instead of cotton wool because cotton wool is attacked by the chemicals).

Note Ensure that there is enough cotton wool in the funnel and that it is tightly wedged into the stem of the funnel so that it fills the opening ensuring that all the liquid has to pass through the cotton wool upon filtration.

Note If you are using string, tie a double knot in it with both knots on top of one another. Then use a pair of scissors to cut the two loose ends of the string off.

4. Place the plastic microburner vial (without the lid) into well F1 in the comboplate and place lid 1 into it.
5. Place the syringe into the larger opening in lid 1.
6. Place a piece of silicone tubing over the smaller opening of lid 1.
7. Place the stem of the microfunnel into the other end of the silicone tubing.
8. Place the plastic microretort stand into well D5 of the comboplate and clamp the silicone tube into the arm just below the stem of the microfunnel so that it is not touching the syringe. This is to hold the funnel upright and to make sure that it is steady during filtration.
9. Make sure that the microfunnel is held as nearly perpendicular as possible so as to minimise spilling of liquid during the filtration.

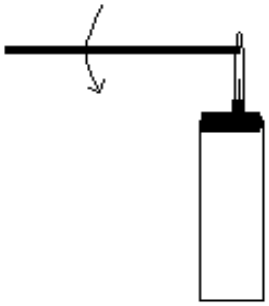
10. Make sure all the connections are sealed tightly otherwise no vacuum will be produced.
11. Slowly pour the solid/liquid mixture in the glass vial into the microfunnel until it is ¾ full.
12. Slowly pull the plunger out of the syringe (this creates a vacuum in the plastic microvial), drawing the liquid out of the microfunnel into the plastic vial leaving the solid residue in the microfunnel.
13. Repeat this process until all the solid has been transferred from the vial to the funnel.
14. Use a glass rod to scrape any solid remaining in the vial into the funnel.
15. You can also use a little water (or suitable solvent) (about 0.1 mℓ) to get any solid that sticks to the sides of the vial into the microfunnel.
16. Having filtered all of the mixture, add a further 0.5 mℓ water (or other suitable solvent) to the original container to wash out into the funnel any crystals that are still adhering to its sides. Filter these washings through the funnel using the syringe. The aim is to get as much of the solid into the funnel as possible.
17. Use a microspatula (or a glass rod) to scrape any remaining crystals out of the original container into the microfunnel.
18. Continue this process until all the solid has been collected in the microfunnel.
19. Some of the original liquid may adhere to the surface of the crystals in the funnel. This can be washed away using a little water (or other suitable solvent), and then continuing the vacuum filtration.
20. Draw up a small amount of water (or other suitable solvent) - about 1 mℓ - into the syringe or a propette.
21. Slowly add it to the funnel containing the solid.
22. Using the suction action of the syringe, draw out this liquid from the funnel.
23. The solid has now been collected and washed.

Note 1 If the plunger is completely pulled out before all of the liquid has been drawn from the microfunnel, remove the syringe from lid 1, and push the plunger completely in. Replace the syringe in lid 1 and draw the plunger out slowly again to continue filtration.

Note 2 The microburner vial may become full, at some point. This means that no more liquid will be suctioned from the funnel. If this is the case, slowly remove the lid from the vial and empty its contents into the organic waste container - make sure you don't lose any crystals in the process - and then replace the lid.

Note 3 Note the difference between ordinary 'gravity' filtration and vacuum filtration. Gravity filtration relies on the force of gravity to draw the liquid through the filter paper into the collection vial. This however, is not always effective, because solid can clog up the filter causing the liquid phase to be unable to pass through the filter paper (ie. the force of gravity is not strong enough to draw the liquid through the filter paper because of the collected solid that clogs it) and the filtration process can take a very, very long time. Vacuum filtration speeds up the process generating an additional force to gravity that draws the liquid through the filter.

3. Making Capillary Tubes for Melting and Boiling Point Determinations



1. Light the microburner flame and hold the one end of a capillary tube horizontally, directly into the flame (see figure 1). The glass should begin to melt and the end of the capillary tube should close.
2. As the glass is melting, twirl the capillary tube between your fingers to make sure it remains straight as it melts.
3. Once the end of the capillary tube is totally sealed, pull it out of the flame and let it cool to room temperature.

4. Boiling Point Determinations

REQUIREMENTS

Apparatus: 1 x Organic comboplate®; lid 1, 1 x glass vial, wire vial-holder, 1 x microburner, matches,1 x paper clip, 2 x elastic bands, 2 x capillary tubes, 1 x 150°C thermometer, 1 x microretort stand, 1 x fusion tube, 1 x propette, 3 capillary tubes, tissue paper.

Chemicals: Boiling chips, paraffin oil, methylated spirits, sample.

PROCEDURE

1. Take a capillary tube sealed at one end. (see General Procedures, section 3).
2. Place lid 1 in well E1 of the comboplate.
3. Assemble the wire vial-holder (see General Procedures, section 1).
4. Place the wire vial holder into the small opening in lid 1 so that it is touching the bottom of the well (see figure 2).
5. Wrap the elastic band around the center of the glass vial, so that it fits tightly.
6. Fill the glass vial with paraffin oil to a depth of approximately 1 cm and place 2-3 boiling chips into the oil.
7. Rest the glass vial in the wire vial-holder.
8. Clip the thermometer into the microretort stand and attach the fusion tube with an elastic rubber band.

9. Place the microretort stand in well D12 and submerge the thermometer and fusion tube halfway into the oil (see figure 3).

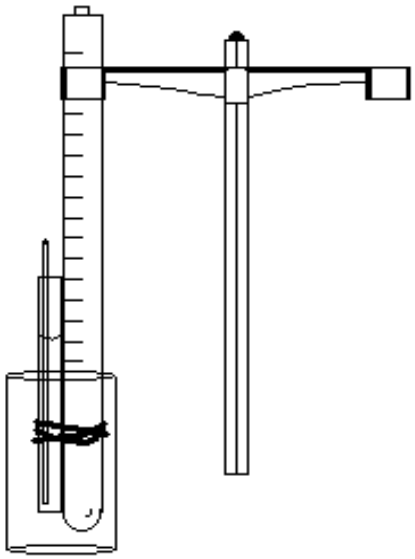


Figure 3

Note Do not let the thermometer or the capillary tube touch the bottom of the vial during heating. Also keep the elastic band out of the oil.

10. Using a propette place the liquid sample into the fusion tube until it is ¾ full.
11. Place the open end of the capillary tube into the fusion tube (see figure 3).
12. Light the microburner and place it directly under the vial (see figure 2). Watch the rise in temperature.
13. Bubbles will emerge from the open end of the capillary tube, slowly at first, but eventually almost continuously. When the stream of bubbles is continuous, stop heating by removing the flame of the microburner from beneath the vial. Extinguish the microburner flame.
14. Observe and record the temperature when the evolution of bubbles just ceases as the liquid cools. This is the boiling point of the liquid. Also note the atmospheric pressure.

Note Do this experiment two times. This is because the boiling temperature is unexpected the first time you conduct the experiment and your first measurement may not be read accurately. Thus, conduct this experiment the first time to obtain a rough estimate, then conduct it again to get a more accurate reading.

15. Take the fusion tube out of the oil and allow it to cool to room temperature.

Note You may have to top up the fusion tube with new sample so that it is ¾ full because some of it may have evaporated in the previous boiling point determination.

16. Keep the rate of temperature rise low for the 2nd determination. This is done by removing the microburner from under the oil every few seconds and replacing it. The closer you get to the boiling point, the slower the rise in temperature should be.

Note When you have completed your 2 observations, discard the capillary tubes. Wash the fusion tube with water, clean out the fusion tube with a piece of tissue paper and leave it in the sun to dry.

5. Melting Point Determinations

REQUIREMENTS

Apparatus: Organic comboplate, lid 1, lid 2, glass vial, wire vial-holder, 1 x microburner, 2 x elastic bands, 2 x capillary tubes, 3 x microspatulas, 1 x microretort stand, 1 x 150°C thermometer.

Chemicals: Boiling chips, paraffin oil, benzoic acid ($C_7H_6O_2$(s)), methylated spirits, unknown solid, matches.

PROCEDURE

1. Prepare 2 capillary tubes for a melting point determination (see General Procedures, section 3)
2. Place 4 microspatulas of finely powdered solid into well A1 of the comboplate.
3. Push the open end of the capillary tube through this portion of the finely powdered solid, making sure that some of the solid is pushed into it.
4. Tap the closed end of the tube on the desk so that the solid collects at the sealed end.
5. A height of solid about 3 mm - 5 mm is suitable.
6. Place lid 1 in well E1 of the comboplate.
7. Wrap the elastic band around the centre of the glass vial, so that it fits tightly.
8. Place the wire vial-holder (see General Procedures, section 1) into the small opening in lid 1 so that it is touching the bottom of the comboplate.
9. Rest the glass vial inside the loop.
10. Fill the glass vial with oil to a depth of approximately 1 cm and place 4-5 boiling chips into the oil to prevent the oil from spluttering out of the vial.
11. Attach the capillary tube to the thermometer using a rubber band (See figure 1). Clip the thermometer into the microretort stand and place it in well D2 of the comboplate.

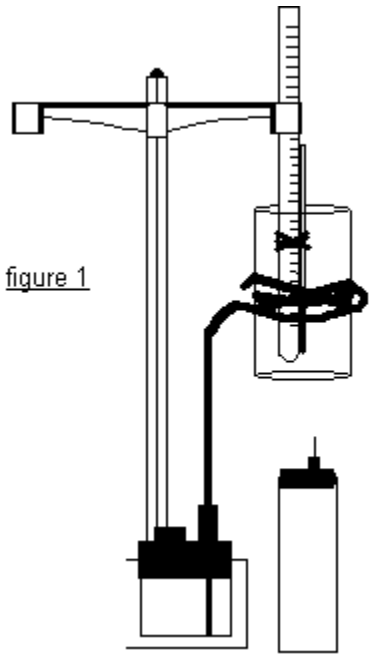


figure 1

12. Submerge the thermometer and capillary tube halfway into the oil.

Note **Do not let the thermometer or the capillary tube touch the bottom of the vial. Do not let the elastic band touch the oil.**

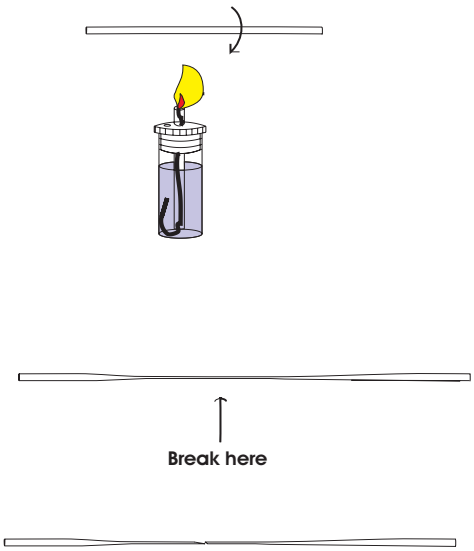
13. Light the microburner and place it directly under the vial.
14. Continue to hold the vial over the flame until the solid melts.
15. Note the temperature at which the solid suddenly melts in the tube.
16. Discard the capillary tube.

Note Repeat this experiment to obtain a second, more accurate result. This is because the melting temperature is reached fairly quickly and your first measurement may not be read accurately. Thus, conduct this experiment the first time to obtain a rough estimate of the melting point. Then conduct it again to get a more accurate result.

17. Keep the rate of temperature rise low for the 2nd determination. This is done by removing the microburner from under the oil every few seconds and replacing it. The closer you get to the melting point, the slower the rise in temperature should be.

6. Making TLC spotting capillary tubes

1. Light the microburner.
2. Hold the middle of a capillary tube about 3 mm over the flame and rotate it with your fingers so that the heat is evenly distributed.
3. The center part of the capillary tube will become supple from the heat.
4. At this point quickly pull it out of the flame and pull the two ends away from each other making the middle part long and thin.



Note Don't hold the tube over the flame too long or it will split in half because of the heat before you have drawn it out.

5. Break the extended tube in half.
6. Each half is a TLC spotting capillary tube.

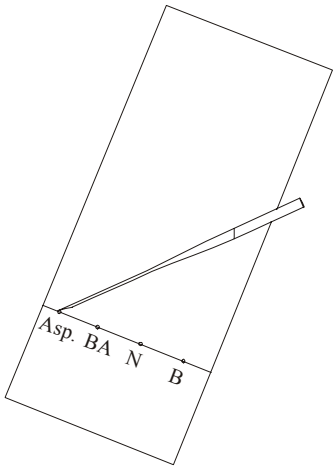
7. A Thin Layer Chromatogram (TLC)

Note **Do not touch the surface of the TLC plate with your fingers. Hold it on it's sides only - like a photographer would hold a photographic plate so as not to get any finger marks on it.**

1. Draw a pencil line across the TLC plate about 10 mm from the bottom.

Note **Do not use an ink pen, the ink will run and make the spots difficult to see.**

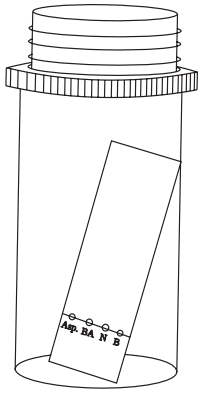
2. Draw as many pencil dots at evenly spaced points across the plate as substances there are to be analysed. These dots are where you spot the TLC plate.
3. Label each dot according to the substance you plan to spot there in pencil.
4. Draw out as many TLC spotting capillaries as spots to be made on the plate (see General Procedures, section 6).
5. Place 1 microspatula of each solid to be spotted into one of the wells of the comboplate.
6. Using a propette, place enough solvent into each well so as to dissolve the solid.
7. Dip a spotting capillary into well E1; it should draw up some of the liquid.
8. Gently dab the end of the capillary onto the TLC plate in the place where you wish the spot to be.



9. Do this for each of the substances you wish to analyse.

Note **Do not make the spot too big by holding the capillary tube against the TLC plate for too long. A diameter of no more than 2 mm is sufficient.**

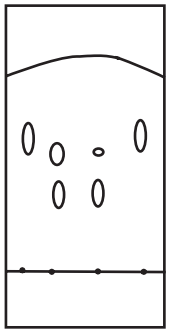
10. Fill the glass vial to a height of about 5 mm with the designated developing solvent (which is normally a mixture of substances).
11. Place the TLC plate inside the vial so that the top is resting against the side of the vial.
12. Place the lid upside down on top of the vial.



13. Allow the developing solvent to run up the TLC plate until it is about ¾ of the way up and then remove the plate from the vial.
14. Immediately trace the developing solvent front with a pencil to indicate where it is. This must be done quickly since the solvent will evaporate and the solvent front won't be visible.
15. Allow the plate to dry for some time.
16. Empty the remaining developing solvent into the waste bottle provided. Not down the sink.
17. Wash the vial with water and dry it with a piece of tissue paper.
18. Put one or two pieces of iodine crystals into the vial and put the lid on properly this time.
19. Pour boiling water into the plastic lunch box and hold the vial over in the steaming water for 1-2 minutes.
20. After a while, the presence of iodine vapour will be observable in the vial.
21. Place your TLC plate into the vial in the same way as before.
22. Brown spots begin to form on the plate corresponding to where the iodine has interacted with the substances spotted.
23. After a minute or two remove the plate from the vial and immediately mark the postion of all spots, by outlining them on the plate with a pencil. This is because the iodine fades after some time.
24. Measure the distance from the line drawn at the bottom of the plate to the midpoint of each spot and record it.
25. Measure the distance of the solvent front from the line drawn at the bottom of the plate to the solvent front directly above each spot.
26. Calculate the Rf value of each spot by dividing distance travelled by the spot by the distance the solvent front travelled.
27. Use a table like the one below to report your results.

Substance	Distance travelled by spot /mm (1)	Distance of solvent front directly above spot /mm (2)	Rf value = (1)/(2)

28. Draw a rough sketch (similar to the one below) of your TLC plate in your report indicating all distances travelled.



CHAPTER 3

Preparation and Reaction of Organic Compounds

Preparation and Reaction of Organic Compounds

PREPARATION AND TESTING OF ETHYNE (ACETYLENE)

In this experiment you will react calcium carbide powder with water to produce a gaseous product called ethyne. Ethyne molecules contain triple bonds. This unsaturation will be tested for using either a potassium permanganate or bromine solution.

REQUIREMENTS

Apparatus: 1 x Organic comboplate®; 1 x lid 1; 1 x lid 2; 1 x syringe; 2 x propettes; 1 x piece of silicone tubing (4 cm x 4 mm); 3 x plastic microspatulas; Gloves (rubber or latex).

Chemicals: Calcium carbide powder ($CaC_2(s)$); Potassium permanganate solution ($KMnO_4(aq)$) [0.1%] or a dilute solution of bromine in water ($Br_2(aq)$); Tap water; Universal indicator solution.

PROCEDURE

Before you begin, ask your teacher about the age of the calcium carbide sample. If the powder is very old or has been exposed to the atmosphere, you may need to remove about 1 cm of the powder from the surface of the sample.

1. Using the spoon of a plastic microspatula, place 3 to 5 spatulas of the calcium carbide powder into well F1 of the organic comboplate®. The quantity used depends on the age of the sample. More than 5 spatulas of an older sample may be needed for the required result to be obtained.
2. Seal well F1 with lid 1.
3. Use a clean propette to fill ⅔ of well F2 with the aqueous solution of potassium permanganate. *(See Question 1)*

Note *You may use a dilute solution of bromine in water as an alternative to the potassium permanganate solution. If the bromine solution is dark brown in colour, place 10 drops of the solution in well F2 and dilute to a pale brown colour using water. The well should not be more than ⅔ full.*

CAUTION! ***Keep the bottles of all bromine solutions sealed during the experiment. Bromine vapours are poisonous!***

4. Seal well F2 with lid 2. Connect wells F1 and F2 by attaching a piece of silicone tubing to the tube outlets on lids 1 and 2 respectively.
5. Fill the syringe with 0,5 mℓ of tap water and fit the syringe into the syringe inlet on lid 1 covering well F1.

Note *Make sure that the wells are sealed tightly, otherwise the ethyne may escape.*

6. Carefully add one drop (about 0,1 mℓ) of water to the calcium carbide powder. If no reaction occurs, gently tap the top of the syringe so that the drop of water falls from the inside of lid 1 onto the powder in the well.

CAUTION! ***Do not add all of the water at once because a violent reaction may occur.*** *(See Question 2)*

7. Add the remainder of the water slowly to the powder in well F1. When no more bubbles are seen in the solution in well F2, remove lid 2 from well F2 and observe the solution in the well. *(See Questions 3 and 4)*

Note *You will probably smell a foul odour and see a white vapour at the vent in lid 2. These phenomena are not due to the ethyne, but are caused by an impurity commonly found in calcium carbide samples.*

8. Remove lid 1 from well F1. Use a clean propette to add one drop of universal indicator solution to the mixture in the well. *(See Question 5)*
9. Use the narrow end of a clean microspatula to stir the changed potassium permanganate solution in well F2. Allow the solution to stand for one to five minutes and observe. (This instruction does not apply where a bromine solution has been used.) *(See Question 6)*

Deposit all organic waste into a waste jar before rinsing the comboplate® and propettes with water.

PREPARATION AND TESTING OF ETHYNE (ACETYLENE)

QUESTIONS

Q1. What is the colour of the potassium permanganate solution?

Q2. What happens in well F1 when a drop of water falls onto the calcium carbide powder?

Q3. What is happening in well F2?

Q4. Has the appearance of the potassium permanganate solution (or bromine solution) changed? Describe.

Q5. Observe the colour of the indicator when added to the mixture in well F1.

Q6. What has happened to the solution in well F2 after about 5 minutes?

Q7. Write down a balanced chemical equation to explain the reaction that occurred in well F1 between the calcium carbide and water.

Q8. Use the answer to question 7 to explain the change in colour of the universal indicator solution in well F1.

Q9. While the gas was bubbling into the solution in F2, the following change took place:

$$\underset{\text{Purple}}{KMnO_4(aq)} \rightarrow \underset{\text{Orange/brown}}{MnO_2(s)}$$

i. Explain what type of reaction this is.
ii. Use the partial equation above to describe why the solution in F2 appeared to change when left for 5 minutes after the reaction was complete.
iii. If a bromine solution was used, write down a balanced chemical equation to show the reaction occurring in well F2. Why was a colour change observed?

Q10. Acetylene gas is highly flammable and gives a very high temperature flame when burned with oxygen. Carbon dioxide and water are produced.
i. Write down a chemical equation to show the reaction of oxygen with acetylene.
ii. Name the process mentioned in question 10.
iii. Identify one industrial use of acetylene gas based on the statement made in question 10.

SYNTHESIS OF ASPIRIN

REQUIREMENTS

Apparatus: 1x Organic comboplate®; 1 x lid 1; 1 x 2.5 mℓ syringe; 2 x propettes; 1 x piece of silicone tubing (4 cm x 4 mm); 2 x plastic microspatula; 1 x microretort stand; 1 x filter funnel; string from the microburner; microburner vial; 1x glass vial (2 cm x 5 cm); glass rod; plastic forceps; scissors; thermometer; lunch box.

Chemicals: Salicylic acid ($C_7H_6O_3$(s)); acetic anhydride ($C_4H_6O_3$(ℓ)); concentrated sulphuric acid (H_2SO_4(conc.)); water.

PROCEDURE

This reaction is exothermic. Don't hold the vial in your hand when adding the acid as it can get too hot to hold and you might drop it. Acetic anhydride is a lachrymator (ie. it makes your eyes 'water'). Upon addition of concentrated sulphuric acid, a very strong, stinging smell is released: make sure the vial is far from your face and try not to breathe in the vapours. Alternatively use a mask to cover your nose and mouth.

1. To the 2 cm x 5 cm glass vial add 6 heaped microspatulas salicylic acid, 0.6 mℓ acetic anhydride using the syringe and 6 drops of concentrated sulphuric acid using a clean propette.
2. Fill the lunch box (or any other suitable container) to about 1/3 full with boiling hot water.
3. Immerse the lower half of the vial into the hot water and swirl it around until all the crystals have dissolved. Continue swirling the reaction mixture for 5 minutes to complete the reaction. If the crystals don't dissolve immediately, use a glass rod to stir the reaction mixture *(see Questions Q1-3)*. Replace the hot water in the lunch box after 2.5 minutes to ensure that a reaction takes place.
4. Empty the hot water from the lunch box, and fill it with ice (or ice cold water).
5. Place the vial in the ice. (If no ice is available, pour cold water into the lunch box - the same volume as the hot water bath).
6. Add 5 mℓ cold water to the reaction vial using the syringe *(see Question 4)*, and cool the contents to about 0°C by leaving it in the ice for 15 minutes. At this point, aspirin crystals should form.

Note *If no crystals separate out after about 5 minutes, scratch the inside of the vial with the glass rod just below the surface of the liquid. Continue cooling until crystals form.*

7. Separate the aspirin crystals by vacuum filtration (see general procedures section 2) and wash the isolated crystals with water *(see Question 5)*. However, instead of cotton wool, use one of the pieces of string from the microburner as the filter plug from the microfunnel (cotton wool is corroded by the chemicals used). Make two knots on top of one another in the string, cut the loose ends off, and force the knot into the stem of the funnel with an unfolded paperclip so that it is totally sealed by the string.
8. Scrape the aspirin out of the microfunnel into well E4 of the comboplate, and store it there overnight to dry. Do not put a lid on the well so that any water left from washing the crystals will evaporate. Do not discard the crystals once collected; keep them for the purification and for testing of purity.
9. Cautiously remove the string from the funnel, scraping any solid that adheres to it into well E4 of the comboplate. Do not undo the knot, but do rinse it with cold water and store it for use in other experiments.

SYNTHESIS OF ASPIRIN

QUESTIONS

Q1. Draw representations of the molecular structure of salicylic acid, acetic anhydride and aspirin.

Q2. Write out a balanced chemical equation for the reaction of salicylic acid with acetic anhydride.

Q3. Explain the use of H_2SO_4 (conc.) in the experiment.

Q4. Acetic anhydride is used in excess in this procedure. This excess can react with the carboxylic acid group in the aspirin product. Which step in the procedure guards against this ? Explain.

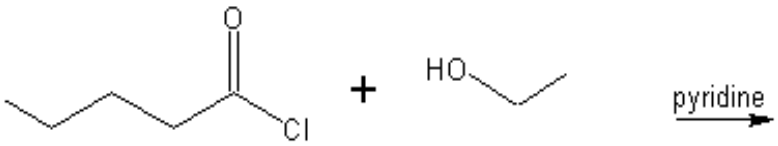
Q5. What is the purpose of washing the crystals ?

EXTENSION QUESTIONS

Q6. If 1 microspatula of salicylic acid has a mass of approximately 0.2 g, calculate the mass of aspirin that should be formed.

Q7. Assuming you obtained 1 g of aspirin product, calculate the percentage yield formed.

Q8. One way to prepare an ester is by the reaction of an alcohol with an acid anhydride (as in this experiment). However, esters also may be prepared by the reaction between an alcohol and a carboxylic acid or an alcohol and an acid chloride. Predict the products of the following reaction:



RECRYSTALLISATION OF CRUDE ASPIRIN AND TEST OF PRODUCT PURITY

In this experiment, crude aspirin crystals are purified by recrystallisation, and tested for impurities with ferric chloride and by a melting point determination.

REQUIREMENTS

Apparatus: Organic comboplate, 1 x lid 1, 1 x 2.5 mℓ syringe; 6 x propettes; 1 x piece of silicone tubing (4 cm x 4 mm); 3 x plastic microspatula; 1 x microretort stand; 1 x filter funnel; string from the microburner; microburner vial; 1 x glass vial (2 cm x 5 cm); glass rod; thermometer; 2 x capillary tubes; 2 x elastic bands.

Chemicals: Crude aspirin product from the Synthesis of Aspirin experiment; pure aspirin; water; crushed ferric chloride crystals ($FeC\ell_3.6H_2O(s)$); paraffin oil.

PROCEDURE

1. Wrap the elastic band around the centre of the vial, so that it fits tightly and place the crude aspirin in it.
2. Add 2 mℓ water and 0.5 mℓ methylated spirits, using the syringe, to your crude aspirin in the vial.
3. Place lid 1 in well F6 of the comboplate.
4. Assemble the wire vial holder (see General Procedures section 1) and the microburner as shown in figure 1.
5. Rest the glass vial in the wire loop.

Note *Adjust the elastic band so that the vial is at least 2 mm above the flame of the microburner.*

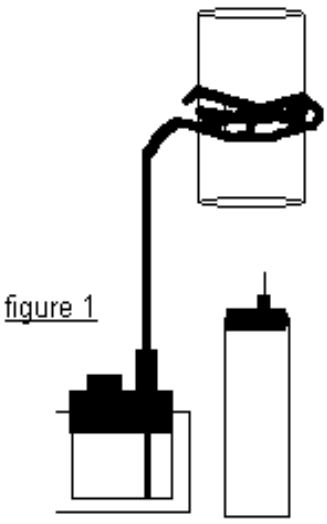


figure 1

6. Place the microburner directly under the vial (see figure 1).
7. Heat the mixture until all the solid has dissolved.
8. When no solid is visible, cease heating and allow the mixture to cool in air to room temperature (this should take about 20 minutes).

Note *If no crystals have formed after 20 minutes, scratch the inside of the vial with a glass rod. This aids crystallisation. If no crystals appear place the vial in ice water.*

9. Vacuum filter the resulting crystals using a clean piece of string and wash with 0.5 mℓ water (see general procedures section 2). Remember to empty the microburner vial of any methylated spirits before using it to collect the filtrate.

10. Using an unfolded paper clip, force the string out of the stem of the funnel and scrape the crystals into well E1 of the comboplate.
11. Leave your recrystallised aspirin to dry in air over 2-3 days *(see Question 1)*.
12. Determine the melting point of (i) pure aspirin (obtained from your teacher) and (ii) your aspirin product after it is dry. First obtain an approximate result and then repeat more slowly to obtain a more accurate result (see General Procedures section 4) *(see Question 2)*. If the dried crystals of your product do not fit into the capillary tube, crush them with a glass rod, until they are fine enough to go into it. You can also use a straightened paper clip to force the crystals into the bottom of the capillary tube.
13. Ferric chloride test: Place one microspatula of your aspirin product into well E5 and 4 microspatulas of pure aspirin into well E6 of the comboplate and fill them both with water.
14. Add a one grain of ferric chloride to each of the reaction mixtures.
15. Stir each of the wells with a glass rod, note the colour of the reaction mixtures and compare them *(see Question 3)*. (If no colour change is observed, add a few more grains of ferric chloride to each well until you observe something).

Note *Make sure you clean the glass rod after stirring so that no impurities from one reaction mixture are transferred to the other, thus contaminating the pure aspirin.*

QUESTIONS

Q1. Why are the crystals dried ?

Q2. Report the melting point of your aspirin product, both the approximate and more accurate observations. Find out the melting point of pure aspirin. How can we use the actual melting point to get an idea of the purity of the aspirin product?

Q3. Report the results of your ferric chloride test on your recrystallised aspirin and on pure aspirin.

Q4. Salicylic acid gives a purple colour with ferric chloride. What does the test show about the presence of salicylic acid in the two aspirin samples?

Q5. Name two possible sources for salicylic acid in your recrystallised aspirin.

Q6. How else could you check the purity of your aspirin product? Explain.

THE OXYACETYLENE FLAME

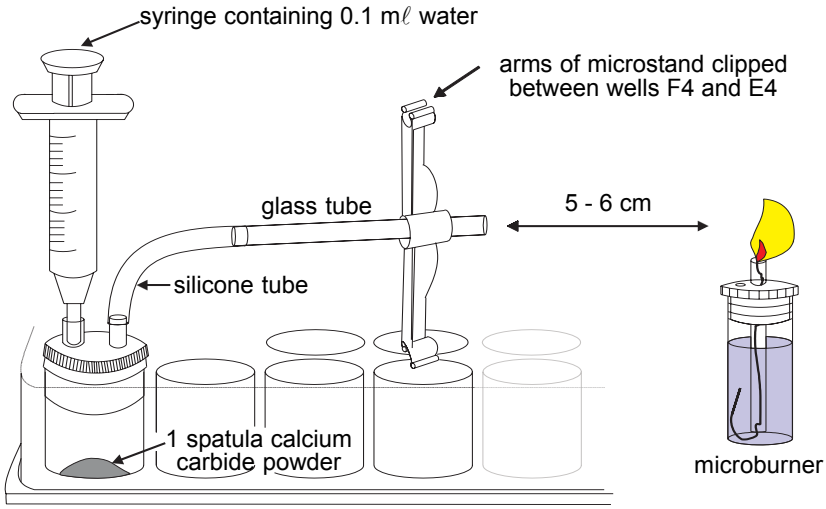
REQUIREMENTS

Apparatus: 1 x Organic comboplate®; 1 x lid 1; 1 x syringe; 1 x piece of silicone tubing (4 cm x 4 mm); 1 x plastic microspatulas; 1 x glass tube; Pair of arms of the plastic microstand; 1 x microburner; Matches; Gloves (rubber or latex).

Chemicals: Calcium carbide powder ($CaC_2(s)$); Tap water; Methylated spirits for the microburner

PROCEDURE

1. Make sure that well F1 is completely dry before using the spoon end of the plastic microspatula to add one level spatula of calcium carbide to well F1. If the sample is old, more spatulas of the calcium carbide may be required (see the experiment on the preparation and testing of ethyne).
2. Seal well F1 with lid 1.
3. Attach the piece of silicone tubing to the tube outlet on lid 1.
4. Join the glass tube to the end of the silicone tubing.
5. Clip one arm of the pair of microstand arms onto the comboplate® between wells F4 and E4 (see diagram below)so that the arms are in an upright position. Make sure that the connection is secure so that the arms do not move or slip during the experiment.



6. Push the glass tube through the central opening in the pair of microstand arms so that it is held in a nearly horizontal position.
7. Assemble the microburner and place it approximately 5 to 6 cm from the open end of the glass tube.
8. Fill the syringe with 0,1 mℓ of tap water and fit the syringe into the syringe inlet in lid 1 covering well F1.
9. Light the microburner. Slowly add the 0,1 mℓ of tap water to the calcium carbide in well F1. Do not add the water too quickly otherwise some of the mixture in F1 will move through the silicone tube into the glass tube. *(See Questions 1 and 2)*

Extinguish the flame of the microburner.
Deposit all organic waste into a waste jar before rinsing the comboplate® and tubes with water.

QUESTIONS

Q1. Observe the end of the glass tube and note what happens.

Q2. Look upwards and around you. Describe what you see in the air.

Q3. Why is the flame in this experiment referred to as an oxyacetylene flame and not an acetylene flame?

Q4. Complete combustion of a hydrocarbon causes the carbon atoms in the hydrocarbon molecule to be oxidised to their highest oxidation state, +4. See the experiment which shows the complete combustion of ethyne.

i. What are the colours of the products of the complete combustion of ethyne?

ii. What was the colour of one of the products you noticed after observing the oxyacetylene flame? Name this product and list its oxidation state.

iii. Why do you think that the product in ii. above was formed?.

ACID-BASE EXTRACTION OF BENZOIC ACID FROM NAPHTHALENE

In this experiment, you will look at a way to separate an organic acid (benzoic acid) from a non-polar organic compound (naphthalene). Both of these substances are soluble in organic solvents and relatively insoluble in water. Since benzoic acid is an acid, we can use this property to separate it from naphthalene by reacting it with a base to form a salt which is soluble in water.

REQUIREMENTS

Apparatus: 1 x Organic comboplate®; 2 x microspatulas, syringe, 1 x glass vial (2 cm x 5 cm) + lid, 5 x propettes, gloves, safety glasses (if available).

Chemicals: Benzoic acid/naphthalene mixture, diethyl ether, water, 2 M NaOH, drying agent (anhydrous sodium sulphate or magnesium sulphate), 2 M HCℓ.

PROCEDURE

Diethyl ether is very flammable and volatile, so keep it way from open flames. It can also be harmful if inhaled, ingested or adsorbed into the skin. So work in a fume hood or a very well ventilated room or even outside.

1. Add 9 microspatulas of the solid mixture (1:1 benzoic acid: naphthalene) to the glass vial.
2. Using the syringe, add 1 mℓ of dethyl ether to the mixture in the glass vial.
3. Place the lid on the vial and swirl it gently until all the solid has dissolved.
4. Clean the syringe with water and use it to add 1 mℓ of 2 M NaOH to the glass vial.
5. Place the lid on the vial and gently shake the vial from side to side for about 1 minute so that the layers are well mixed.

Note This step causes the NaOH to be able to react with the benzoic acid. The product of this reaction is a salt that is soluble in the aqueous phase. *(see Questions 1 & 2 & 8)*

6. Place the vial on your desk and leave the two layers to separate for about 1 minute.
7. Place the comboplate directly next to the vial, as close to well F6 as possible.
8. Expel the air from a propette and dip it into the solution so that it rests against the bottom of the vial and draw up only the lower aqueous layer. As quickly as possible, remove the propette from the vial and release the solution into well F6 of the comboplate (if necessary, repeat the process until all the lower layer is removed).

Note This step must be done as quickly as possible since the liquid may squirt out of the end of the propette. Also, don't allow the top of the propette to inflate fully once you have depressed it; keep it depressed slightly all the time while transferring. This stops any squirting out of the nozzle.

9. Add 1 microspatula of drying agent to the vial and leave the mixture to dry for 5 minutes. *(see Question 3)*
10. Place a clean propette into the vial and make sure that it is resting against the bottom of the vial. Draw up as much of the liquid as you can without drawing up any drying agent. Place the liquid into well F1 of the comboplate leaving the solid drying agent behind. Allow it to evaporate in the sun (or in the wind) leaving the solid naphthalene in the well. This can take about 30 minutes.
11. Clean the vial and transfer the aqueous phase, still in well F6 of the comboplate, to the vial using a clean propette.
12. Add 1 mℓ of 2 M HCℓ to the vial using the syringe. Put the lid on and shake it gently to mix the reagents. The solution should go cloudy because of the formation of white benzoic acid solid. *(see Questions 6 & 9)*

13. Clean the syringe and add 1 mℓ ether to the vial, place the lid on the vial and shake for a minute to extract the precipitate into the ether layer. *(see Question 5)*

    The solution should go clear again because the solid dissolves in ether.

14. Expel the air from a clean propette and dip it into the solution so that it rests against the bottom of the vial and draw up the lower aqueous layer and place it into well E6 of the comboplate.

15. Add 2 microspatulas of drying agent and leave the mixture to dry for 5 minutes. *(see Question 3)*

16. Place a clean propette into the vial and make sure that it is resting against the bottom of the vial. Draw up as much of the liquid as you can without drawing up any drying agent. Place it into well F3 of the comboplate. Allow it to evaporate in the sun (or in a drafty place) leaving the benzoic acid solid in the well *(see Question 10)*. This can take about 30 minutes. *(see Question 4)*

ACID-BASE EXTRACTION OF BENZOIC ACID FROM NAPHTHALENE

QUESTIONS

Q1. Why do we shake the reaction vial?

Q2. Which layer does the benzoic acid salt go into?

Q3. Why do we add drying agent to the first layer transferred to the comboplate ?

Q4. What does the word 'decant' mean? In which steps of the experiment, could we have decanted a liquid?

Q5. Which is the ether layer - explain why !

Q6. What is the precipitate formed upon the addition of HCℓ?

Q7. Draw out the line drawings of benzoic acid and naphthalene.

Q8. Write out a balanced chemical equation for the reaction that occurs in step 5 of the procedure.

Q9. Write out a balanced chemical equation for the reaction that occurs in step 12 of the procedure.

Q10. After the liquid has evaporated, what substance do you see in well F1 of the comboplate? And E1?

SAPONIFICATION (making soap)

REQUIREMENTS

Apparatus: Glass vial (2 cm x 5 cm), silicone tubing, microfunnel, comboplate, 1 x 2 mℓ syringe, 3 x microspatulas, wooden stick, lid 1, microburner, glass rod, microretort stand, silicone tubing, wire vial holder.

Chemicals: Boiling chips, butter, salt, water, sodium hydroxide solution (2 M NaOH(aq)), methylated spirits, universal indicator paper, commercial soap, calcium chloride solution (1 M $CaC\ell_2$(aq)).

PROCEDURE

1. Wrap a piece of elastic band around the center of the glass vial, so that it fits tightly.
2. Place 10 microspatulas of butter in the bottom of the glass vial. *(see Question 1)*
3. Use the syringe to place 8 mℓ of the 2 M NaOH into the glass vial.
4. Add 3-4 boiling chips to reduce bumping.
5. Place lid 1 on well F6 of the comboplate.
6. Place the wire vial holder in the small opening in lid 1
7. Rest the glass vial in the wire loop of the wire vial holder.
8. Place the microburner directly under the vial, and adjust the elastic band so that the bottom of the vial is about 3 mm above the microburner.
9. Light the microburner and heat the mixture until it starts to boil gently.
10. After the first 10 minutes of heating, add another 1 mℓ of 2 M NaOH to the solution (this is because some of it will have evaporated). Continue heating until the mixture begins to boil again, and then extinguish the microburner flame.

Note *If the mixture boils vigorously and the level of the liquid starts to rise because of boiling, remove the flame from under the vial for 20 seconds and then replace it.*

11. Add ice to the lunch box (if no ice is available, use cold water).
12. Place the vial and its contents into the ice bath, add 1 microspatula of salt to the solution and dissolve it by stirring the mixture. Cool the mixture for 15 minutes in ice. Two layers should separate out, a flaky hard organic soap layer and an aqueous alcohol layer. *(see Question 3)*
13. **Vacuum filtration and washing:** Separate the soap from the liquid using the technique outlined in the general procedures section 2 (use some string with a double knot in it as the filter frit) and wash it with about 1 ml ice cold water.
14. Unclip the funnel from the retort stand and using a glass rod, scrape the filtered solid into well F2 of the comboplate.
15. **pH tests:** Into well F3 of the comboplate add one microspatula of your soap and fill the well with water. Stir it with a glass rod to make sure that some of the soap dissolves in the water.
16. Test the pH of the solution using a strip of universal indicator paper and a glass rod. Dip the glass rod into the reaction mixture and then touch the indicator paper with the glass rod to determine the pH of the solution *(see Question 5)*.
17. Do the same with the commercial soap provided, making up a solution of water and soap in well F4 of the comboplate.
18. **Reaction with hard water** *(see Question 4)*: Into wells E5 and F5 of the comboplate, add tap water using a syringe until each are ¾ full.
19. Adding 8 drops of calcium chloride solution to well F5 using a propette. This causes the water to become 'hard'.

20. Add a small portion of soap (about 2 microspatulas) to wells F5 & E5 and stir it vigorously with a glass rod until the soap has dissolved. Observe and compare the two solutions. (*See Question 4*)

Note *Make sure the glass rod is clean before stirring both solutions. It can transfer impurities from the one solution to the other.*

21. **Soap in salt water:** Clean and dry the glass vial and wrap the elastic band around the center.

22. Place one microspatula of your soap and 1 mℓ water into the glass vial.

23. Stir the mixture vigorously with a glass rod and then heat it over the microburner using the wire vial holder until all the soap has dissolved.

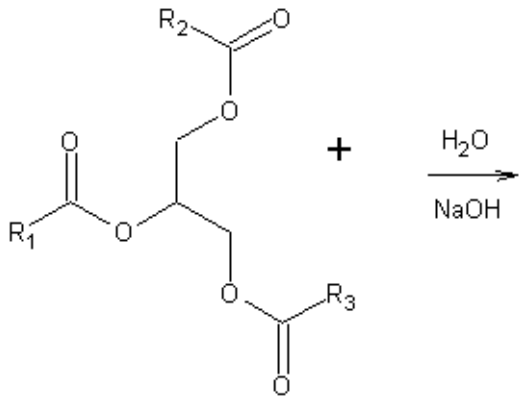
24. Stop heating the mixture and let it cool to room temperature.

25. Remove the vial from the holder and add 2 microspatulas of salt and stir the mixture. Note what happens. *(see Question 6)*

SAPONIFICATION (making soap)

QUESTIONS

1. Butter is an ester of glycerol with butyric (butanoic) acid and other carboxylic acids.
   - Write out the chemical formula and the line drawing for glycerol.
   - Write out the chemical formula and line drawing for butyric acid.
   - Propose a chemical structure for the glycerol tributyrate ester.
   - Write out a balanced chemical equation for the following reaction and identify the soap.



   - (optional) Draw out the reaction mechanism for the reaction above.

2. Name some other household products that contain fats that could be used in this experiment instead of butter.

Properties of soap

3. Why is the soap layer the upper layer and the aqueous layer the lower layer ?
4. What is the colour of the universal indicator paper ? What is the pH ? Explain your observations.

	Crude Soap Product	Commercial Soap
pH reading with universal indicator paper		

5. What did you observe when you added the soap to the hard and soft water ? Explain your observations.

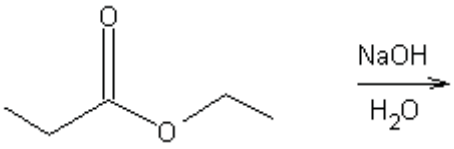
	Normal Tap Water	Normal Tap Water + Calcium Chloride Solution = Hard Water
Observation (after stirring the soap solution vigorously)		

6. Note what you observe when you add salt to the soap solution, and explain it.

EXTENSION QUESTIONS

7. What is the cause of the cleansing properties of soap? (Hint: look up "micelles" in an organic chemistry textbook).

8. Predict the product of the following reaction:



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