Cambridge Assessment International Education

Teaching Pack

Identifying alkenes, alcohols and halogenoalkanes

Cambridge International AS & A Level Chemistry 9701

Version 1.0

Cambridge Pathway

Contents

Icons used in this pack:

Briefing lesson

Planning lesson

Lab lesson

Debriefing lesson

Introduction

This pack will help you to develop your learners' experimental skills as defined by assessment objective 3 (AO3 Experimental skills and investigations) in the course syllabus.

Important note
Our *Teaching Packs* have been written by **classroom teachers** to help you deliver topics and skills that can be challenging. Use these materials to supplement your teaching and engage your learners. You can also use them to help you create lesson plans for other experiments.

This content is designed to give you and your learners the chance to explore practical skills. It is not intended as specific practice for Paper 3 (Advanced Practical Skills) or Paper 5 (Planning, Analysis and Evaluation).

This is one of a range of *Teaching Packs* and each pack is based on one experiment. The packs can be used in any order to suit your teaching sequence.

The structure is as follows:

Briefing lesson (1 hour*)
This lesson reinforces the key concepts, relevant skills, knowledge and understanding linked to the experiment.

↓

Planning lesson (1 hour*)
This lesson focuses on planning an experiment.
It ends with a demonstration video of the experiment.

↓

Lab lesson (1 hour*)
This lesson focuses on carrying out the experiment including the collection and recording of observations, measurements and estimates.

↓

Debriefing lesson (1 hour*)
This lesson focuses on the analysis and interpretation of data.
This includes making conclusions, evaluating methods and the quality of data and how improvements could be made.

** the timings are a guide only; you may need to adapt the lessons to suit your circumstances.*

In this pack you will find lesson plans, worksheets and teacher resource sheets.

Experiment: Identifying alkenes, alcohols and halogenoalkanes

There are a vast number of organic compounds which have a similar physical appearance yet contain different functional groups. This pack enables learners to identify the following functional groups: alkenes, alcohols and halogenoalkanes.

This experiment has links to the following syllabus content (see syllabus for detail):

- 14.2 Alkenes
- 16.1 Alcohols
- 15.1 Halogenoalkanes
- 22.1-22.2 Analytical techniques

The experiment covers the experimental skills, as listed in **AO3: Experimental skills and investigations:**

- plan experiments and investigations
- collect, record and present observations, measurements and estimates
- analyse and interpret data to reach conclusions
- evaluate methods and quality of data and suggest improvements.

Prior knowledge

Learners will most probably have met these functional groups before in IGCSE Chemistry and also be able to draw and name simple aliphatic analogues.

Briefing lesson: Simple organic molecules

Resources	• Worksheet A • Molecular models (if available)

Learning objectives	By the end of the lesson: • ***all*** learners should be able to recognise some of the features of the given compounds • ***most*** learners should be able to recognise the majority of the features of the given compounds • ***some*** learners will be able to recognise all of the features of the given compounds.

Timings	Activity
10 min	**Starter/Introduction** Ask some of your learners to draw the structural formulae of any **alkane** on the board. Look at the variety of structures drawn and discuss why there are so many possibilities. Depending on what the learners remember about organic chemistry, the conversation may involve discussions about crude oil, fractional distillation and isomerism.
10 min	**Main lesson** Discuss the meaning of the term 'functional group' with your learners and ask them which ones they can recall. Ask learners to draw a map of the different functional groups found in organic chemistry. This will form the basis of a single sheet that will depict the different functional groups that learners need to know about and that can be added to with additional information such as the tests to distinguish them and how to change one to another. This will be useful when learners come to revise this topic.
20 min	Hand out Worksheet A. If available, it is strongly encouraged that learners build models of the molecules to help them. If molecular models are unavailable, there are 3D models provided within the *Resource Plus* platform.
10 min	Learners should check each other's answers here using the answer sheet provided.
10 min	**Plenary** Ask the learners, in pairs, to imagine they were given a number of colourless organic liquids to identify. By considering qualitative analysis tests for inorganic compounds, learners should try to make a brief list of reasons how qualitative analysis for inorganic compounds is quite different to that for organic compounds. If the learners are unable to think of any, the following could be discussed: • tests for inorganic compounds are based on the fact that they contain ions. In organic compounds, there are no ions to test. • Tests for organic compounds usually only serve to identify certain functional groups in the molecules, whereas, in inorganic tests, it is often possible to identify the actual compound (cation(s) and anion(s)). • Organic liquids are often flammable, so the use of Bunsen burners is prohibited.

Planning lesson: Identifying colourless liquids

Resources	• Worksheet B • Small samples of lithium chloride, sodium chloride and potassium chloride • Colourless organic liquid samples

Learning objectives	By the end of the lesson: • ***all*** learners should be able to identify the functional groups to be tested and the names of the tests for each • ***most*** learners should be able to suggest why a specific test is used for a particular functional group • ***some*** learners will be able to describe each test in detail, explain why it is used and make links between the tests for organic compounds and inorganic compounds.

Timings	Activity
10 min	**Starter/Introduction** Show your learners three solids: lithium chloride, sodium chloride and potassium chloride (no labels). Ask them what type of tests they would do to find out the identity of the solids (allow the discussion to explore cation and anion identification). Then give learners more information by giving the compound names – based on this new information, what is the main difference (the cation) and how can these cations be distinguished (allow discussion of flame tests).
10 min 30 min	**Main lesson** Provide learners with Worksheet B. Show them some samples of colourless organic liquids. The liquids are labelled as follows: • **1A** and **1B** (cyclohexane / cyclohexene) • **2A** and **2B** (methanol / ethanol) • **3A** and **3B** (1-bromobutane / 1-iodobutane) • **4A** and **4B** (propan-1-ol / 2-methylpropan-2-ol) Explain that the aim of the experiment is to find a chemical test that will help to identify each liquid pair. Ask learners to draw each of the structures in the pairs in their lab book. If some learners struggle with this, they can skip it and go onto section two of the worksheet. You may need to provide assistance to learners for some of the structures. Next, learners should use the prompts on the worksheet to record the required information for each pair of substances. They should: • identify the functional group in each pair • identify the test required for this functional group • explain why this test gives a positive result • explain what is involved in the test • note any safety and disposal considerations for each pair. An example of what is required is given on the worksheet.
10 min	**Plenary** Watch the experiment video which shows the relevant tests. Allow time for learners to update their lab books and make any final adjustments to their planning.

Lab lesson: Identifying organic liquids

Resources	• *Teacher walkthrough* video • Worksheet C	

Learning objectives	By the end of the lesson: • ***all*** learners should be able to correctly identify some of the liquids • ***most*** learners should be able to correctly identify most of the liquids • ***some*** learners will be able to correctly identify all of the liquids.

Timings	Activity
10 min	**Starter/Introduction** Discuss the different types of hazards associated with working with the materials required for this experiment. Show some of the hazard symbols for the materials in these tests. Can they identify the labels? Discuss other safety concerns such as why would the water-baths be prepared using hot water from a kettle? Remind learners of the safe disposal methods associated with working with organic liquids.
40 min	**Main lesson** Learners work in pairs. During the planning lesson, many learners may have chosen suitable tests to discern between the pairs of colourless liquids. In doing so, they may have suggested different amounts of reagents to use and slightly varying conditions. In the interests of management of resources, the amounts and concentration of reagents have been specified. **Safety** Circulate the classroom at all times during the experiment so that you can make sure that your learners are safe and that the data they are collecting is accurate. Hand out Worksheet C to the learners. This sheet provides the scaffold for learners to complete the work in the lab books. To avoid congestion, ask learners to start with different tests. **Safety** For Pair 1 and Pair 3, it is advisable to work in a fume cupboard. If fume cupboards are not available, then these experiments can be performed carefully in a well-ventilated area. Suggestions for how to clear up after each experiment might have been determined by learners during the planning phase. However, do provide this information, which is provided for you in the pack, to learners.
10 min	**Plenary** Learners should now reflect on the tests that they have done. Did they see the expected results for each test to allow them to confidently distinguish between each pair of colourless liquids? Ask if any learners saw something unexpected and what they think might be the issue.

Teacher notes

Watch the *Identification of alkenes, alcohols and halogenoalkanes* (*Teacher Walkthrough*) and read these notes.

Each pair will require access to:

- cyclohexane (**1A**)
- cyclohexene (**1B**)
- absolute ethanol (**2A**)
- methanol (**2B**)
- 1-iodobutane (**3A**)
- 1-bromobutane (**3B**)
- propan-1-ol (**4A**)
- 2-methylpropan-2-ol (**4B**)
- bromine water [0.005 mol dm^{-3}]
- sodium hydroxide solution [1.0 mol dm^{-3}]
- iodine solution [0.5 mol dm^{-3}] in potassium iodide solution [0.2 mol dm^{-3}]
- silver nitrate solution [0.1 mol dm^{-3}]
- deionised water
- acidified potassium dichromate(VI) solution [0.2 mol dm^{-3}]
- clean test-tubes
- one beaker (250 cm^3) per group to make a water-bath
- water-baths set at approx. 50 °C and 80 °C [*if unavailable, electric kettle(s), for learners to make up hot water-baths themselves using glass beakers*]
- standard lab alcohol thermometers
- plastic pipettes
- 2 × bungs per pair
- Heavy metals disposal bottle

Safety

The information in the table below is a summary of the key points you should consider before undertaking this experiment with your learners.

It is your responsibility to carry out an appropriate risk assessment for this experiment.

Substance	Hazard	First aid
Cyclohexane	 GHS02 (*flammable* **F**)  GHS07 (*moderate hazard* **MH**)	**In the eye:** flood the eye with gently-running tap water for 10 min and consult a doctor. **Swallowed:** wash out the mouth with water. Do not induce vomiting. Consult a doctor. **Spilt on skin or clothing**: remove contaminated clothing and shoes immediately and rinse. Wash off the skin with plenty of water. Consult a doctor. **Clothing catches fire:** smother flames on clothing or skin with a fire blanket or other

Substance	Hazard	First aid
	GHS08 (*health hazard* **HH**) GHS09 (*hazardous to the aquatic environment* **N**)	material. Cool any burnt skin with gently-running tap water for 10 min. **Other fires**: allow fires in sinks, etc. to burn out. Fires at the top of test-tubes, beakers, etc. should be smothered with a damp cloth or heat-proof mat. **Spilt on floor, bench, etc.:** put out all Bunsen burner flames. Wipe up small amounts with a cloth. Rinse well. For larger amounts open windows, cover with mineral absorbent (e.g. cat litter), scoop into a bucket and add water.
Cyclohexene	GHS02 (*flammable* **F**) GHS07 (*moderate hazard* **MH**) GHS08 (*health hazard* **HH**) GHS09 (*hazardous to the aquatic environment* **N**)	**In the eye:** flood the eye with gently-running tap water for 10 min and consult a doctor. **Swallowed:** wash out the mouth with water. Do not induce vomiting. Consult a doctor. **Spilt on skin or clothing**: remove contaminated clothing and shoes immediately and rinse. Wash off the skin with plenty of water. Consult a doctor. **Clothing catches fire:** smother flames on clothing or skin with a fire blanket or other material. Cool any burnt skin with gently-running tap water for 10 min. **Other fires**: allow fires in sinks, etc. to burn out. Fires at the top of test-tubes, beakers, etc. should be smothered with a damp cloth or heat-proof mat. **Spilt on floor, bench, etc.:** put out all Bunsen burner flames. Wipe up small amounts with a cloth. Rinse well. For larger amounts open windows, cover with mineral absorbent (e.g. cat litter), scoop into a bucket and add water.
Methanol	GHS02 (*flammable* **F**) GHS07 (*moderate hazard* **MH**) GHS08 (*health hazard* **HH**)	**In the eye:** flood eye with gently-running tap water for 10 min. Consult a doctor. **Swallowed:** do no more than wash out the mouth with water. Do not induce vomiting. Sips of water may help cool the throat and help keep the airway open. See a doctor. **If inhaled:** move person into fresh air. If not breathing, give artificial respiration. Consult a doctor. **Clothing catches fire:** smother flames on clothing or skin with fire blanket or other material. Cool any burnt skin with gently-running tap water for 10 min. **Other methanol fires:** allow fires in sinks, etc. to burn out. Fires at the top of test-

Substance	Hazard	First aid
		tubes, beakers, etc. should be smothered with a damp cloth or heat-proof mat. **Spilt on floor, bench, etc.:** put out all Bunsen burner flames. Wipe up small amounts with a cloth. Rinse well. For larger amounts open windows, cover with mineral absorbent (e.g. cat litter), scoop into a bucket and add water. **Spilt on the skin or clothing**: remove contaminated clothing. If more than a test-tube amount was involved, wash the affected area and clothing with plenty of water.
Ethanol	 GHS02 (*flammable* **F**)  GHS07 (*moderate hazard* **MH**)  GHS08 (*health hazard* **HH**)	**In the eye:** flood the eye with gently-running tap water for 10 min. Consult a doctor. **Swallowed:** do no more than wash out the mouth with water. Do not induce vomiting. Sips of water may help cool the throat and help keep the airway open. Consult a doctor. **Note:** The person may show signs of drunkenness. **Spilt on the skin or clothing:** remove contaminated clothing and rinse it. Wash the affected area and clothing with plenty of water. **Clothing catches fire:** Smother flames on clothing or the skin with a fire blanket or other material. Cool any burnt skin with gently-running tap water for 10 min. **Other ethanol fires:** Allow fires in sinks, etc. to burn out. Fires at the top of test-tubes, beakers, etc. should be smothered with a damp cloth or heat-proof mat. **Spilt on the floor, bench, etc.:** extinguish all Bunsen burner flames. Wipe up small amounts with a cloth and rinse it well. For larger amounts, open all windows, cover with mineral absorbent (e.g. cat litter), scoop into a bucket and add water.
1-Bromobutane	 GHS02 (*flammable* **F**) 	**In the eye:** immediately flush the eyes with plenty of water for at least 15 min, occasionally lifting the upper and lower eyelids. Consult a doctor. **Swallowed:** do not induce vomiting. Consult a doctor. **If inhaled:** remove from exposure and move to fresh air immediately. If not breathing, give artificial respiration. If

Substance	Hazard	First aid
	GHS08 (*health hazard* **HH**)	breathing is difficult, give oxygen. Consult a doctor. **Spilt on skin or clothing:** immediately flush skin with plenty of water for at least 15 min while removing contaminated clothing and shoes. Consult a doctor.
1-Iodobutane	 GHS02 (*flammable* **F**)  GHS08 (*health hazard* **HH**)	**In the eye:** immediately flush the eyes with plenty of water for at least 15 min, occasionally lifting the upper and lower eyelids. Consult a doctor. **Swallowed:** do not induce vomiting. Consult a doctor. **If inhaled:** remove from exposure and move to fresh air immediately. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Consult a doctor. **Spilt on skin or clothing:** immediately flush skin with plenty of water for at least 15 min while removing contaminated clothing and shoes. Consult a doctor.
Propan-1-ol	 GHS02 (*flammable* **F**)  GHS07 (*moderate hazard* **MH**)	**In the eye:** flood the eye with gently-running tap water for 10 min. See a doctor. **Swallowed:** do no more than wash out the mouth with water. Do not induce vomiting. Sips of water may help cool the throat and help keep the airway open. See a doctor. **Vapour breathed in:** move person into fresh air. Keep them warm. See a doctor if breathing is difficult. **Clothing catches fire:** smother flames on clothing or the skin with a fire blanket or other material. Cool any burnt skin with gently-running tap water for 10 min. **Other fires:** allow fires in sinks, etc. to burn out. Fires at the top of test-tubes, beakers, etc. should be smothered with a damp cloth or heat-resistant mat. **Spilt on the skin or clothing:** remove contaminated clothing. If more than a test-tube amount was involved, wash the affected area and clothing with plenty of water. **Spilt on the floor, bench etc.:** put out all Bunsen burner flames. Wipe up small amounts with a cloth and rinse it well. For larger amounts, open all windows, cover with mineral absorbent (e.g. cat litter), scoop into a bucket and add water.
2-Methylpropan-2-ol		**In the eye:** flood the eye with gently-running tap water for 10 min. See a doctor. **Swallowed:** do no more than wash out the mouth with water. Do not induce vomiting.

Substance	Hazard	First aid
	GHS02 (*flammable* **F**)  GHS07 (*moderate hazard* **MH**)	Sips of water may help cool the throat and help keep the airway open. See a doctor. **Vapour breathed in:** move person into fresh air. Keep them warm. See a doctor if breathing is difficult. **Clothing catches fire:** smother flames on clothing or the skin with a fire blanket or other material. Cool any burnt skin with gently-running tap water for 10 min. **Other fires:** Allow fires in sinks, etc. to burn out. Fires at the top of test-tubes, beakers, etc. should be smothered with a damp cloth or heat-resistant mat. **Spilt on the skin or clothing:** remove contaminated clothing. If more than a test-tube amount was involved, wash the affected area and clothing with plenty of water. **Spilt on the floor, bench etc.:** put out all Bunsen burner flames. Wipe up small amounts with a cloth and rinse it well. For larger amounts, open all windows, cover with mineral absorbent (e.g. cat litter), scoop into a bucket and add water.
Bromine water [0.005 M]	 GHS07 (*moderate hazard* **MH**)	**In the eye:** flood the eye with gently-running tap water for 10 min. See a doctor. **Swallowed:** do no more than wash out the mouth with water. Do not induce vomiting. Sips of water may help cool the throat and help keep the airway open. See a doctor. **Vapour breathed in:** move the person to fresh air. Call a doctor if breathing is even slightly affected. **Spilt on the skin or clothing:** for liquid bromine or the moderately-concentrated solution, immerse in sodium thiosulfate solution (20%, 1 mol dm^{-3}). Remove contaminated clothing, soak it and drench the skin with plenty of water. See a doctor. **Spilt on the floor, bench, etc.:** for spills of all but a few drops of liquid bromine, open windows and evacuate the laboratory. For small bromine spills, add sodium thiosulfate solution (20%, 1 mol dm^{-3}) and leave for 1 h. Mop up and rinse with plenty of water
Sodium hydroxide solution [1 mol dm^{-3}]	 GHS05 (*corrosive* **C**)	**In the eye:** flood the eye with gently-running tap water for at least 20 min. See a doctor. If a visit to hospital is necessary, continue washing the eye during the journey in an ambulance. **Swallowed:** do no more than wash out the mouth with water. Do not induce vomiting.

Substance	Hazard	First aid
		Sips of water may help cool the throat and help keep the airway open. See a doctor. **Spilt on the skin or clothing:** remove contaminated clothing. Drench the skin with plenty of water. If a large area is affected or blistering occurs, see a doctor. **Spilt on the floor, bench, etc.:** wipe up small amounts with a damp cloth and rinse it well. For larger amounts, and especially for (moderately) concentrated solutions, cover with mineral absorbent (e.g. cat litter) and scoop into a bucket. Neutralise with citric acid. Rinse with plenty of water.
Iodine solution [0.5 mol dm^{-3}] in potassium iodide solution [0.2 mol dm^{-3}]	 GHS07 (*moderate hazard* **MH**)  GHS09 (*hazardous to the aquatic environment* **N**)	**In the eye:** flood the eye with gently-running tap water for 10 min. See a doctor. **Swallowed:** do no more than wash out the mouth with water. Do not induce vomiting. Sips of water may help cool the throat and help keep the airway open. See a doctor. **Vapour breathed in:** move the person to fresh air. Call a doctor if breathing is even slightly affected. **Spilt on the skin or clothing:** brush off solid iodine and immerse in sodium thiosulfate solution (20%, 1 M). Remove contaminated clothing, soak it and drench the skin with plenty of water. See a doctor if a large area is affected or blistering occurs. **Spilt on the floor, bench, etc.:** scoop up any solid iodine, add sodium thiosulfate solution (20%, 1 M) to the remaining spill and leave for 1 h. Mop up and rinse with plenty of water.
Silver nitrate solution [0.1 mol dm^{-3}]	 GHS05 (*corrosive* **C**)  GHS09 (*hazardous to the aquatic environment* **N**)	**In the eye:** flood the eye with gently-running tap water for at least 10 min. See a doctor. **Swallowed:** do no more than wash out the mouth with water. Do not induce vomiting. Sips of water may help cool the throat and help keep the airway open. See a doctor. **Spilt on the skin or clothing:** remove contaminated clothing and rinse it. Wash off the skin with plenty of water. If the silver nitrate produces more than small burns, see a doctor. **Spilt on the floor, bench, etc.:** wear eye protection and gloves. Scoop up the solid. Rinse the area with water and wipe up, rinsing repeatedly. Rinse the mop or cloth thoroughly.

Substance	Hazard	First aid
Acidified potassium dichromate(VI) solution [0.2 mol dm^{-3}]	 GHS03 (oxidising **O**)  GHS08 (*health hazard* **HH**)  GHS09 (*hazardous to the aquatic environment* **N**)	**In the eye:** flood the eye with gently-running tap water for at least 10 min. See a doctor. **Swallowed:** do no more than wash out the mouth with water. Do not induce vomiting. Sips of water may help cool the throat and help keep the airway open. See a doctor**.** **Spilt on the skin or clothing:** remove contaminated clothing and rinse it until no colour remains. Wash off the skin with plenty of water. If skin contamination is more than small, see a doctor. **Spilt on the floor, bench, etc.:** wear eye protection and gloves. Scoop up the solid. Rinse the area with water and wipe up, rinsing repeatedly until no colour remains. Rinse the mop or cloth thoroughly.

Teacher method

This is your version of the method for this experiment that accompanies the *Teacher walkthrough* video.

Do not share this method with learners. Give them Worksheet C or Worksheet D.

Before you begin

Plan how you will group your learners during the experiment session.

Think about:

- if there is enough equipment and space for the learners to work in pairs (if not, increase the group size accordingly)
- the amount of equipment/chemicals required. Each pair/group does not require exclusive access to the reagents in this experiment. Arrange the samples and reagents so that several groups can gain easy access.
- Groups should not all start working with pair 1. Organise it such that learners start working on different pairs of chemicals to identify.
- If there are no fume cupboards, explain that learners should set up experiments 1 and 3 in well-ventilated areas of the lab.

Experiment

In this experiment, a variety of chemicals are to be used by the learners. Please refer to the safety information above about each chemical.

Water-baths will need to be ready at approximately 50 °C for pair 3 and at 80 °C for pair 4. If you do not have water-baths, use an electric kettle to boil water and the learners can adjust the temperature of their own water-baths, made using glass beakers.

Given that many of the chemicals are flammable, ensure that there are no matches or Bunsen lighters left within reach of the learners.

Walk around the learners during the experiment in case they encounter any difficulties.

Steps	Notes
1. Check that learners have access to all of the equipment and chemicals they need for this experiment.	*Ensure that they do experiments 1 and 3 in the fume cupboard or a well-ventilated area.*
2. **Pair 1: cyclohexane (1A) and cyclohexene (1B)** – the test for unsaturation	*Make sure gloves are used in this experiment. Make sure that learners do not take the cyclohexene out of the fume cupboard or leave the top off. It has a very powerful odour.*

3. Approximately 0.5 cm^3 of each of samples 1A and 1B are placed in separate test-tubes

It is sufficient to use a plastic pipette for this. Ensure learners replace the bottle tops and leave the pipettes in the fume cupboard so the residual chemicals evaporate.

4. Add the bromine water to each tube and agitate.

If no reaction occurs in the tube containing the alkene (1B) it is probably because the learners did not agitate the tube. You may need to advise learners how to agitate the tube from side-to-side gently, rather than needing to find a bung and shake violently.

5. **Pair 2: ethanol (2A) and methanol (2B)** – the iodoform test.

6. Add 10 drops of each sample into clean test-tubes.

7. Then add 25 drops of iodine solution to each tube.

8. Finally, 10 drops of sodium hydroxide solution are added to each tube. Swirl gently and wait for 2 minutes.

Sample 2A should produce a positive test. A yellow precipitate of tri-iodomethane (iodoform) should be produced. Learners may notice the characteristic 'medical' smell produced in the positive test which you can explain that is due to the presence of tri-iodomethane.

9. **Pair 3: 1-iodobutane (3A) and 1-bromobutane (3B)** – test for halide ions

It may be worth discussing the analogous test used in inorganic chemistry with halides and silver nitrate. Learners would have encountered this at IGCSE.

10. To two clean test-tubes, add 0.5 cm^3 of 0.1 mol dm^{-3} silver nitrate (95% ethanol solution).

Prepare a solution of 0.1 mol dm^{-3} $AgNO_3$ in 95% ethanol for learners to use. A water-bath will need to be ready at approx. 50 °C.

11. Add four drops of sample 3A and 3B.

12. Loosely place a bung in the test-tubes and place in the water bath at 50 °C.

Despite this explicit instruction on their worksheets, be sure to check that none of the bungs have been placed tightly in the mouth of the test-tubes.

13. Allow time for the precipitates to develop.

For sample 3A, the yellow precipitate should develop quickly. It may take more time for the cream-coloured precipitate to develop with sample 3B.

14. Dispose of the contents of the test-tubes.

Even though the amounts used are very small, ensure that learners dispose of the contents of the test-tubes responsibly, by placing in the heavy metal recovery bottle.

15. **Pair 4: propan-1-ol (4A) and 2-methylpropan-2-ol (4B)** – oxidation

16. Place the samples (approx. 0.5 cm^3) in clean test-tubes.

17. Add approx. 1 cm^3 of the acidified potassium dichromate(VI) solution to each tube.

18. Place both samples in the hot water bath at 80 °C and leave for about 5 min.

Sample 4A should turn green fairly quickly. If it has not after 5 min, advise learners to leave in the bath a little longer. Sample 4B is a tertiary alcohol, so no oxidation should occur and thus the orange colour of the unchanged potassium dichromate(VI) solution should remain.

19. Dispose of the contents of the test-tubes.

Again, even though the amounts used are very small, ensure that learners dispose of the contents of the test-tubes responsibly, by placing in the heavy metal recovery bottle.

Clean-up

In addition to having placed the potassium dichromate(VI) and silver residues in a separate collection bottle, learners should:

- clean all glassware
- tidy up their work space
- ensure any spillages have been mopped up
- return all equipment and any unused chemicals to you.

Debriefing lesson: Use of analytical techniques

Resources	• Worksheets E, F, G and H • Suggested answers for Worksheet E

Learning objectives	By the end of the lesson: • ***all*** learners should be able to explain some differences between the analytical data for the pairs of molecules • ***most*** learners should be able to explain many of the differences between the analytical data for the pairs of molecules • ***some*** learners will be able to comprehensively show the differences between the analytical data for the pairs of molecules.

Timings	Activity
10 min	**Starter/Introduction** '*In the qualitative analysis of inorganic compounds, it is often possible to determine the exact formula of a test substance. However, regarding organic substances, it is often not possible to do this*.' Ask learners, in pairs, to discuss this statement for a couple of minutes and feedback thoughts to the class. Next, in their lab books, ask learners to make a brief list of methods that are commonly used nowadays to determine the exact structures of organic molecules. [It is hoped that learners will be prompted to suggest and then briefly discuss a number of analytical techniques including mass spectrometry, carbon-13 and ^{1}H (proton) NMR, and infra-red spectroscopy.
40 min	**Main lesson** Give Worksheet E, Worksheet F, Worksheet G and Worksheet H to pairs of learners. With the exception of a slight change to the third pair of molecules, these are the same as those used in the previous main practical lesson. Learners should choose which of the following analytical techniques would be the most helpful in discerning between the pairs of molecules: • mass spectrometry • infra-red • carbon-13 NMR • ^{1}H (proton) NMR spectroscopy. With guidance, learners should be able to apply a number of the syllabus learning points to this exercise (22.2–22.5).
10 min	**Plenary** Learners compare their suggested solutions to those given in the suggested answers.

Worksheets and answers

Worksheet A: Alkenes, alcohols and halogenoalkanes

Here are six organic compounds.

A: $CH_3CH_2CH_2OH$

B: $CH_3CH_2C(OH)(CH_3)CH_3$

C: $CH_3(CH_2)_3CH_3$

D: $CH_3CH{=}CHCH_3$

E: $BrCH_2CH_2CH_3$

F: $CH_3CH(OH)CH_2CH_3$

1. In your lab book:
    a. Draw each of the structures **A–F**
    b. Identify the functional group
    c. Write the name of the compound

You are strongly encouraged to build models of each of the structures before you draw them.

2. There are **three** different classes of alcohols. Label the alcohols in your list with the correct class.

3. These three different classes can also be used for halogenoalkanes. Draw structures of each class of halogenoalkanes and name the compounds. You may choose any halogen atom.

4. One of the qualitative tests for cations and anions could be adapted to determine the functional group of which structure?

5. (a) What kind of reactions do alkenes undergo?

   (b) Name **three** simple molecules that would react with ethene.

Worksheet B: Experiment design

Use this worksheet to inform what you write in your lab book.

You have been provided with the following **four** pairs of colourless organic liquids.

- **Pair 1:** cyclohexane and cyclohexene
- **Pair 2:** methanol and ethanol
- **Pair 3:** 1-bromobutane and 1-iodobutane
- **Pair 4:** propan-1-ol and 2-methylpropan-2-ol

The compounds within each pair will be labelled as either **A** or **B**.

Your task is to choose a suitable qualitative test to perform on each pair of liquids.

1. Draw the displayed formulae for each compound.

2. For each pair:
   a. identify the functional group
   b. identify the test required
   c. explain why the test will give the result expected
   d. describe how to carry out the test, including equipment and reagents
   e. describe any safety and disposal precautions required.

Example: propanone and propanal
Functional group(s) present ketone = RCOR' aldehyde = RCOH (both contain a carbonyl group C=O)
Name of the test required Tollens' silver mirror test.
Explanation of why test is required This test gives a positive result with aldehydes but not with ketones.
Description of the test When an aldehyde is warmed with an ammoniacal solution of silver ions, a silver mirror is formed on the inside of the test tube.
Safety and disposal: Flush contents down the sink with plenty of water.

Worksheet C: Experiment guidance

Use this sheet as guidance when writing your results in your lab book.

You have been supplied with four pairs of colourless organic liquids. You are required to perform appropriate tests on each compound and from your results decide on the identity of each sample.

For each pair, you should record in your lab books the following information:

- the reagents required
- the equipment required
- safety information
- your procedure and conditions
- your results (observations)
- your conclusions
- disposal methods.

Worksheet D: Further experiment guidance

Pair 1: cyclohexane and cyclohexene	
Reagents	Bromine water (0.005 mol dm^{-3} solution).
Equipment	Two test-tubes, plastic pipettes.
Safety	Gloves should be worn and this test should be performed **in a fume cupboard**. Cyclohexane and cyclohexene are both highly flammable and irritating to the eyes, skin and lungs.
Procedure and conditions	Place approximately 0.5 cm^3 of each of the samples **1A** and **1B** into separate clean test-tubes. Next add 2–3 drops of bromine water to each test-tube and agitate gently.
Disposal	Allow the small amount of volatile compounds to evaporate in the back of the fume cupboard. Your teacher will remove these tubes later.

Pair 2: methanol and ethanol	
Reagents	Methanol, absolute ethanol, sodium hydroxide (1 mol dm^{-3}), iodine solution (0.5 mol dm^{-3}) in potassium iodide solution (0.2 mol dm^{-3}).
Equipment	Test-tubes, plastic pipettes.
Safety	Methanol and ethanol are highly flammable. Additionally, methanol is highly toxic. Sodium hydroxide solution is corrosive.
Procedure and conditions	Add 0.5 cm^3 of sample **2A** to a clean test-tube. Next add 1 cm^3 of the iodine solution followed by 0.5 cm^3 of the sodium hydroxide solution. Gently swirl the test-tube. Wait for 2 min and observe any changes. Repeat this procedure for sample **2B**.
Disposal	The small quantities used in the experiment can safely be flushed down the sink with plenty of water.

Pair 3: 1-bromobutane and 1-iodobutane	
Reagents	1-Bromobutane, 1-iodobutane, ethanol, silver nitrate solution (0.1 mol dm^{-3}), water, sodium hydroxide.
Equipment	Test-tubes with bungs (placed loosely), a hot water-bath, a glass beaker, plastic pipettes.
Safety	Both 1-bromobutane and 1-iodobutane are considered hazardous and must be used **in a fume cupboard**. Gloves should be used when handling. Ethanol is flammable. Silver nitrate at this low concentration is considered relatively safe.
Procedure and conditions	Set up a water-bath at around 50 °C. To a clean test-tube add 0.5 cm^3 of 0.1 mol dm^{-3} silver nitrate solution in 95% ethanol. Then add four drops of sample **3A**. Loosely place a bung in the test tube and place in the water-bath. After 5 min note the colour of the precipitate formed. Repeat this procedure using sample **3B**.
Disposal	Place all silver containing residues in a heavy metals or silver recovery bottle.

Worksheet D: Further experiment guidance, continued

Pair 4: propan-1-ol and 2-methylpropan-2-ol	
Reagents	Propan-1-ol and 2-methylpropan-2-ol, acidified potassium dichromate solution (0.2 mol dm^{-3}).
Equipment	Test-tubes, hot water-bath set at 80 °C.
Safety	Potassium dichromate(VI) is highly toxic and oxidising and is dangerous for the environment.
Procedure and conditions	Place sample **4A** (0.5 cm^3) in a clean test-tube. Add the acidified potassium dichromate(VI) solution (1 cm^3). Place the tube in a hot water bath at 80 °C and wait for 5 min. Repeat this procedure with sample **4B**.
Disposal	Do not throw this waste down the sink. Collect it in a bottle for recovery/disposal.

Worksheet E: Use of analytical techniques

Mass spectrometry, infra-red spectroscopy, carbon-13 NMR and ^{1}H (proton) NMR spectroscopy are all commonly used techniques to analyse organic molecules.

It is common to use several of these together, in order to confirm a particular structure.

Consider the following pairs of organic compounds:

- cyclohexane and cyclohexene
- methanol and ethanol
- 1-bromobutane and 1-chlorobutane
- propan-1-ol and 2-methylpropan-2-ol.

As in the previous practical exercise, imagine that these pairs of colourless liquids were unlabelled.

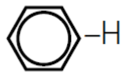
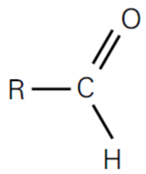
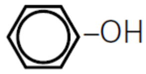
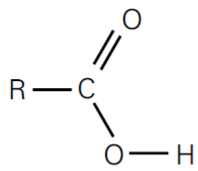
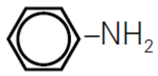
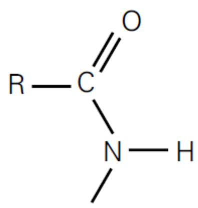
Your task is to show how a combination of analytical techniques could be used to discern between each of them.

You will need to use the supplied data sheets to help you answer these questions in your lab book.

Once again, it is strongly recommended that you draw the displayed formulae for each of these compounds or build models to help you.

Worksheet F: ^{1}H NMR chemical shifts

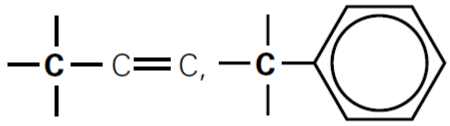
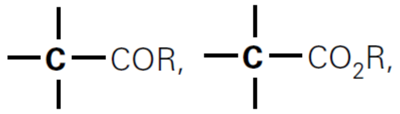
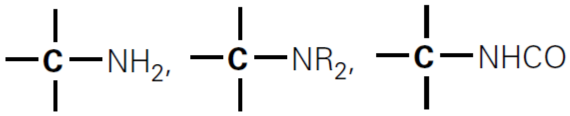
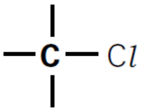
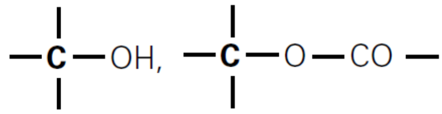
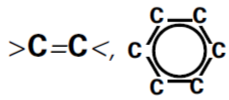
These chemical shift values (δ) are relative to TMS = 0

Type of proton	Environment of proton	Example structures	Chemical shift range (δ)
C–H	alkane	$–CH_3$, $–CH_2–$, >CH–	0.9–1.7
	alkyl next to C=O	$CH_3–C=O$, $–CH_2–C=O$, >CH–C=O	2.2–3.0
	alkyl next to aromatic ring	$CH_3–Ar$, $–CH_2–Ar$, >CH–Ar	2.3–3.0
	alkyl next to electronegative atom	$CH_3–O$, $–CH_2–O$, $–CH_2–Cl$, >CH–Br	3.2–4.0
	attached to alkyne	≡C–H	1.8–3.1
	attached to alkene	$=CH_2$, =CH–	4.5–6.0
	attached to aromatic ring		6.0–9.0
	aldehyde		9.3–10.5
O–H (see note below)	alcohol	RO–H	0.5–6.0
	phenol		4.5–7.0
	carboxylic acid		9.0–13.0
N–H (see note below)	alkyl amine	R–NH–	1.0–5.0
	aryl amine		3.0–6.0
	amide		5.0–12.0

Note: δ values for O–H and N–H protons can vary depending on solvent and concentration.

Worksheet G: Carbon-13 (^{13}C) NMR chemical shifts

These chemical shift values (δ) are relative to TMS = 0

Hybridisation of the carbon atom	Environment of carbon atom	Example structures	Chemical shift range (δ)
sp^3	alkyl	**C**H_3–, **C**H_2–, –**C**H<, >**C**<	0–50
	next to alkene/arene		10–40
	next to carbonyl/carboxyl		25–50
	next to nitrogen		30–65
	next to chlorine (-– CH_2-Br and –CH_2-I are in the same range as alkyl)		30–60
	next to oxygen		50–70
sp^2	alkene or arene		110–160
	carboxyl	R–**C**O_2H, R–**C**O_2R	160–185
	carbonyl	R–**C**HO, R–**C**O–R	190–220
sp	alkyne	R–**C**≡**C**–	65–85
	nitrile	R–**C**≡N	100–125

Worksheet H: IR absorption frequencies

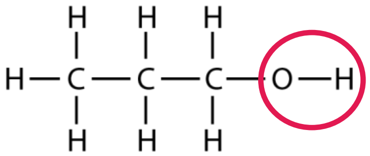
Here are some characteristic infra-red absorption frequencies for some selected bonds.

Bond	Functional groups containing the bond	Absorption range (in wavenumbers) / cm^{-1}	Appearance of peak (***s*** = ***strong***, ***w*** = ***weak***)
C–O	alcohols, ethers, esters	1040–1300	***s***
C=C	aromatic compounds, alkenes	1500–1680	***w*** unless conjugated
C=O	amides	1640–1690	***s***
	ketones and aldehydes	1670–1740	***s***
	carboxylic acids	1680–1730	***s***
	esters	1710–1750	***s***
C≡C	alkynes	2150–2250	***w*** unless conjugated
C≡N	nitriles	2200–2250	***w***
C–H	alkanes, CH_2–H	2850–2950	***s***
	alkenes/arenes, =C–H	3000–3100	***w***
N–H	amines, amides	3300–3500	***w***
O–H	carboxylic acids, RCO_2–H	2500–3000	***s*** and very broad
	H-bonded alcohol, RO–H	3200–3600	***s***
	free alcohol, RO–H	3580–3650	***s*** and sharp

Worksheet A: Answers

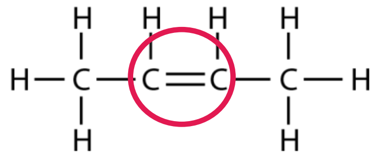
1.

A



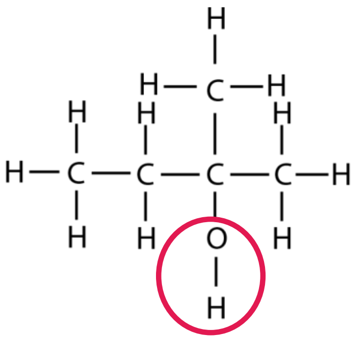
propanol

D



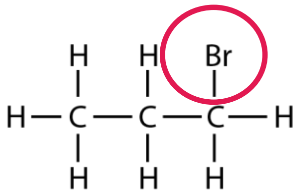
but-2-ene

B



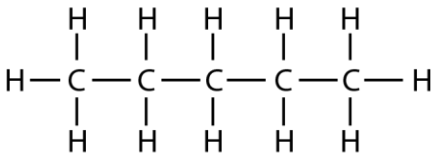
2-methylbutan-2-ol

E



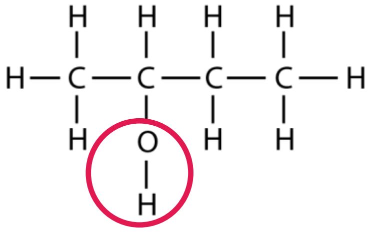
1-bromopropane

C



pentane

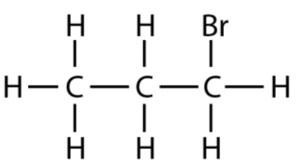
F



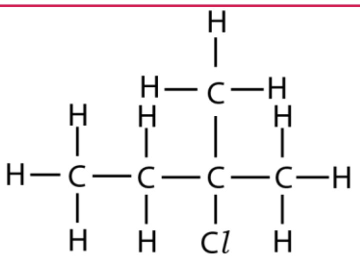
butan-2-ol

2. A primary; B tertiary; F secondary

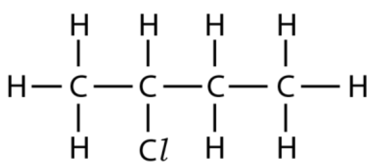
3. You may have chosen any halogen atom for this question. Example answers include:



1-bromopropane



2-chloro-2-methylbutane



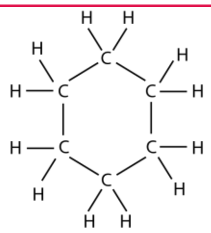
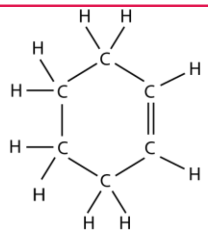
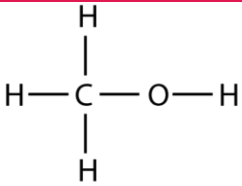
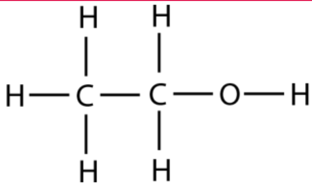
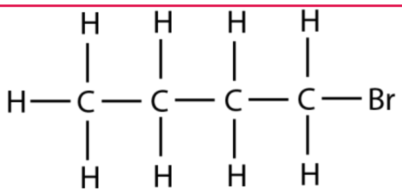
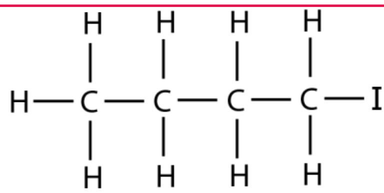
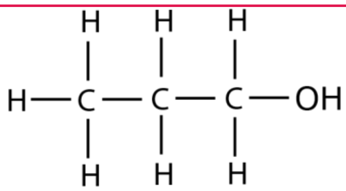
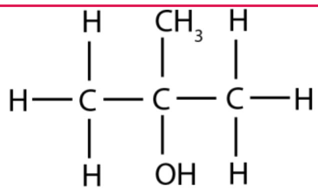
2-chlorobutane

4. The test for a halide ion could be adapted to determining the functional group in the halogenoalkanes, E. (Circled above in question 1.)

5. (a) addition reactions
(b) Some possibilities are: hydrogen, water, bromine and hydrogen bromide.

Worksheet B: Answers

1.

Pair 1: cyclohexane and cyclohexene		
Pair 2: methanol and ethanol		
Pair 3: 1-bromobutane and 1-iodobutane		
Pair 4: propan-1-ol and 2-methylpropan-2-ol		

2.

Pair 1: cyclohexane and cyclohexene
Functional group(s) present: *None in the alkane and a C=C double bond in the alkene.*
Name of the test required: *Test for unsaturation using aqueous bromine water.*
Explanation of why test is required: *In the case of the unsaturated alkene, the bromine will become decolourised. Decolourisation will not occur with the alkane.*
Description of the test: *2 – 3 drops of bromine water is added to the test substance (0.5 cm^3) and the test-tube gently agitated.*
Safety and disposal: *Allow the small amount of volatile compounds to evaporate in the back of the fume cupboard.*

Worksheet B: Answers, continued

Pair 2: methanol and ethanol
Functional group(s) present: *Both are alcohols containing the –OH group.*
Name of the test required: *The iodoform test.*
Explanation of why test is required: *Ethanol will give a positive iodoform test, causing iodoform (tri-iodomethane) to be formed as a yellow precipitate. Methanol will not give this result.*
Description of the test: *Add 10 drops of the test substance to a clean test-tube. Next, add 25 drops of the iodine solution followed by 10 drops of the sodium hydroxide solution. Gently swirl the test-tube. Wait for 2 min and observe any changes.*
Safety and disposal: *The small quantities used in the experiment can safely be flushed down the sink with plenty of water.*

Pair 3: 1-bromobutane and 1-iodobutane
Functional group(s) present: *Both are primary halogenoalkanes containing a carbon–halogen bond.*
Name of the test required: *Test for halogenoalkanes.*
Explanation of why test is required: *Depending on the nature of the halogen, different colours of precipitates are formed.*
Description of the test: *To a clean test-tube add 0.5 cm^3 of silver nitrate in 95% ethanol. Then 4 drops of the halogenoalkane are added. After 5–10 min in a water-bath at 50 °C, note the colour of the precipitate formed. 1-Iodobutane will produce a yellow precipitate (rapidly) whereas, the bromo analogue will (more slowly) produce a cream-coloured precipitate.*
Safety and disposal: *Place all silver containing residues in a heavy metals or silver recovery bottle.*

Worksheet B: Answers, continued

Pair 4: propan-1-ol and 2-methylpropan-2-ol
Functional group(s) present: Both are alcohols containing the –OH group.
Name of the test required: Oxidation of primary (and secondary) alcohols using acidified potassium dichromate(VI) solution.
Explanation of why test is required: 2-Methylpropan-2-ol is a tertiary alcohol and will not undergo oxidation under normal conditions. Propan-1-ol is a primary alcohol and will readily undergo oxidation to give first the aldehyde and finally the acid. A characteristic change from orange to green will be observed.
Description of the test: Pipette the test alcohol (0.5 cm^3) into a clean test-tube. Add potassium dichromate(VI) solution (1 cm^3). Place the tube in a hot water-bath at 80 °C and wait for 5 min.
Safety and disposal: Since chromium is a heavy metal, put the residues in a bottle for recovery/disposal.

Worksheet E: Answers

FOR ALL OF THESE ANSWERS, PLEASE REFER TO THE COPIES OF THE ACTUAL SPECTRA OF THESE COMPOUNDS. IT IS IMPORTANT THAT LEARNERS REALISE THAT NOT ALL OF THE PEAKS CAN BE EASILY IDENTIFIED AT THIS LEVEL.

Pair 1: cyclohexane and cyclohexene

Infra-red

In cyclohexene there is a characteristic C=C double bond peak at 1500–1680 cm^{-1}.

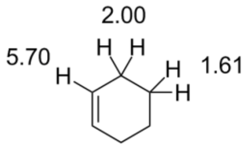
Mass spectrometry

This would be useful to determine the relative formula mass of each compound by finding the mass ion peak for each. Cyclohexane gives a mass ion peak of m/z 84 whereas cyclohexene will give a mass ion peak at m/z 82.

Proton NMR

Cyclohexane will produce a singlet (at about δ 1.5 ppm) due to the twelve equivalent protons in its structure.

Cyclohexene, however, will produce a spectrum with three peaks due to the three different environments of the protons within the molecule. If the area under the peaks was inspected (by integration), the ratio of the number of protons under each peak (reading left to right below) would be: 2:4:4



C-13 NMR

Cyclohexane will again produce a singlet peak since all six carbon atoms are in the same environment.

In cyclohexene, there are three different environments for the carbon atoms giving three peaks in the C-13 spectrum. The peak at 127 ppm corresponds to the two carbon atoms of the double bond. There are two peaks quite close together at 22 ppm and 25 ppm. These are due to the other four carbon atoms and the carbons responsible for the peak at 22 ppm are those furthest from the influence of the double bond.

Worksheet E: Answers, continued

Pair 2: methanol and ethanol
Infra-red *This technique will not be useful here as both molecules contain the same functional group (R–OH).*
Mass spectrometry *This would be useful to determine the relative formula mass of each compound by finding the mass ion peak for each.* *Methanol has a mass ion peak for CH_3OH^+. Analysis of the fragmentation patterns for methanol reveals that the large peak at m/z 31 is due to methanol losing one proton to give the CH_3O^+ ion. Loss of oxygen atom leaves CH_3^+ ion at m/z 15.* *For ethanol the m/z peak is at 46. Loss of one proton produces the $CH_3CH_2O^+$ peak at m/z 45.* **Note:** More advanced learners may identify the CH_2OH^+ peak at *m/z* 31 and the CH_3^+ peak at *m/z* 15.
Proton NMR *Methanol has a singlet at about 3.4 ppm. This corresponds to the three equivalent H atoms of the methyl group. The singlet at approx. 4.0 ppm is due to the de-shielded proton of the –OH group.* *Ethanol shows two alkyl peaks. The $–CH_3$ protons are at 1.2 ppm. Since they are adjacent to a CH_2 group, they produce a triplet splitting pattern. The $–CH_2–$ protons are coupled to the $–CH_3$ protons and produce a quartet splitting pattern.* **Note:** Depending on the ability level of your class you may wish to discuss the splitting pattern and position of the –OH group proton. You may wish to discuss point 22.5e of the syllabus here (identifying OH protons by D_2O exchange).
C-13 NMR *Methanol produces a singlet at about 50 ppm whereas ethanol gives rise to two peaks due to the two different environments of the carbon atoms. The $–CH_2–$ group is attached to an oxygen atom that causes it to be more downfield at approx. 58 ppm. The $–CH_3$ is correspondingly higher field at about 18 ppm.*

Worksheet E: Answers, continued

Pair 3: 1-bromobutane and 1-chlorobutane
Infra-red There would be no significant difference between the two spectra.
Mass spectrometry This would be useful to determine the relative formula mass of each compound by finding the mass ion peak for each. Learners should be familiar with the M+2 peak (syllabus ref. 22.3c).
Proton NMR Not very helpful in discerning between the two analogues, but useful in confirming that the compounds are indeed both primary halogenobutanes in that they both contain: • A triplet at about 3.5 ppm. This is for the $-CH_2-$ next to the halogen atom (C1). The influence of the halogen atom is strongest at C1, thus causing these protons to appear the most downfield. • A quintet at about 1.7 ppm which is the $-CH_2-$ adjacent to that carrying the halogen atom (C2). • The hydrogen atoms attached to C3. This appears as a multiplet (sextet) at about 1.3 ppm. • The terminal carbon, C4, at about 0.8 ppm is a triplet (coupled to two protons).
C-13 NMR The chemical shifts of C2, C3 and C4 are in very similar positions in both molecules. However, since chlorine is much more electronegative than bromine, the position of C1 is significantly different. Thus, in 1-chlorobutane C1 is at approx. 45 ppm and in 1-bromobutane it is at approx. 35 ppm.

Worksheet E: Answers, continued

Pair 4: Propan-1-ol and 2-methylpropan-2-ol

Infra-red

This technique would not be useful since both molecules contain the same functional group.

Mass spectrometry

For propan-1-ol, as expected, there is a molecular ion at m/z 60. If learners have not considered the molecule further, it would be worth discussing the base ion at m/z 31 which corresponds to the CH_2OH^+ species.

For 2-methylpropan-2-ol there is a molecular ion peak at m/z 74 as predicted. However, for reasons beyond the scope of this course, it is very small. There is also a strong peak at m/z 31 which corresponds to the CH_2OH^+ ion.

Proton NMR

For propan-1-ol, we have the following peaks:

- The $-CH_2-$ methylene protons (on C1) are most influenced by the $-OH$ group and appear at approx. 0.9 ppm. They are coupled to an adjacent methylene and thus are seen as a triplet.
- The two protons attached to C2, are coupled to a terminal methyl group and also a $-CH_2-$ group. This gives rise to a multiplet at 1.5 ppm.
- The peak at 3.5 ppm, is a triplet, representing the terminal methyl group, coupled to a $-CH_2-$ group.
- The $-OH$ has given rise to a singlet in this spectrum but depending on the conditions can vary in its position and multiplicity.

For 2-methylpropan-2-ol, since all of the protons attached to the central carbon atom in the molecule are equivalent, the spectrum is a single peak at about 1.2 ppm, which if integrated would show that it represents nine protons.

C-13 NMR

For propan-1-ol, we have the following peaks:

- The $-CH_2-$ adjacent to the $-OH$ group is most de-shielded and is at about 63 ppm
- The terminal carbon has a peak at approx. 10 ppm.
- The mid-chain carbon atom gives rise to a peak at about 25 ppm.

In 2-methylpropan-2-ol, three carbon atoms are in the same environment giving rise to a large peak at approx. 30 ppm. The peak for the remaining carbon atom, attached to the $-OH$ group is considerably downfield at approx. 70 ppm.

Cambridge Assessment International Education
The Triangle Building, Shaftesbury Road, Cambridge, CB2 8EA, United Kingdom
t: +44 1223 553554   f: +44 1223 553558
e: info@cambridgeinternational.org   www.cambridgeinternational.org