Cambridge Assessment International Education

Teaching Pack

Identifying aldehydes and ketones

Cambridge International AS & A Level Chemistry 9701

Version 1.0

Cambridge Pathway

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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 aldehydes and ketones

This *Teaching Pack* focuses on the analysis of organic molecules. In inorganic qualitative analysis it is often possible to fully identify a compound by its constituent anions and cations. In contrast, with organic molecules, analysis focuses on attempting to identify different functional groups within a molecule. To elucidate a structure completely, spectroscopic techniques must be used.

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

- 13.1 Formulae, functional groups and the naming of organic compounds
- 17.1 Aldehydes and ketones

The experiment covers the following 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

None but it is strongly recommended that learners do the following *Teaching Pack* before attempting this one 'Identifying alkenes, alcohols and halogenoalkanes'.

Briefing lesson: Carbonyl containing compounds

Resources	• Worksheet A • Molecular models (if available)
Learning objectives	By the end of the lesson: • ***all*** learners should be able to identify, draw and name some of the relevant carbonyl containing compounds • ***most*** learners should be able to identify, draw and name most of the relevant carbonyl containing compounds • ***some*** learners will be able to identify, draw and name all of the relevant carbonyl containing compounds

Timings	Activity
10 min	**Starter/Introduction** Learners should be divided into pairs for this starter. In their lab books ask them to write one example structure of an aldehyde and a ketone (any aldehyde or ketone). Ask learners to identify the similarities and differences between them. Follow this with a brief classroom discussion, which should identify that: • they both contain a carbonyl group C=O • aldehydes have at least one hydrogen atom attached to the carbon atom carrying the double bond. (If learners suggest methanal, it would be worth mentioning that in this case, there are two hydrogen atoms attached to the carbon atom carrying the double bond.)
10 min 20 min 10 min	**Main lesson** Explain the importance of being able to recognise functional groups in organic compounds especially when they are written as condensed formulae. Learners should work in pairs. Hand out Worksheet A. The object of this exercise is to be able to further identify carbonyl containing compounds and identify which are aldehydes and ketones. 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 that you can project in class. Particular care should be taken where functional groups appear written on the left-hand side of a molecule. This sometimes causes difficulty in understanding which atoms are attached to which. Learners should check each other's answers using the answers provided.
10 min	**Plenary** Explain briefly to learners that they are going to concentrate on the chemistry of aldehydes and ketones for the rest of the activities in this pack. Ask them if they can remember any tests to distinguish between aldehydes and ketones.

Planning lesson: Identifying aldehydes and ketones

Resources	• Worksheet B • Molecular models (if available)
Learning objectives	By the end of the lesson: • ***all*** learners should be able to draw some of the structures and suggest suitable tests to discern between some pairs of compounds • ***most*** learners should be able to draw many of the structures and suggest suitable tests to discern between some pairs of compounds • ***some*** learners will be able to draw all of the structures and suggest suitable tests to discern between each pair of compounds.

Timings	Activity
10 min	**Starter/Introduction** Ask learners if they can recall how to distinguish between an alkene and alkane using chemical tests. Ask what chemicals are needed and what the observed results would be.
10 min	**Main lesson** Learners should already have an understanding of the qualitative analysis of organic molecules from the pack entitled 'Identifying alkenes, alcohols and halogenoalkanes'. Provide learners with Worksheet B. Show them some samples of the pairs of colourless organic liquids. The details can be found in the teacher method.
30 min	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 this 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.
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 aldehydes and ketones

Resources	• *Teacher walkthrough* video • Worksheet C • Worksheet D (as required)
Learning objectives	By the end of the lesson: • **all** learners should be able to identify some of the pairs of liquids • ***most*** learners should be able to identify most of the pairs of liquids • ***some*** learners may be able to identify all of the pairs of 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 hot 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. Worksheet D may be handed out to learners who need more support or were unable to suggest either some or all of the tests required for this practical exercise. Suggestions for how to clear up after each experiment might have been determined by learners during the planning phase. However, do provide this information to learners. It is provided for you in this pack.
10 min	**Plenary** Learners should now reflect on the tests that they have done. Did they see the expected results for each test 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 identifying aldehydes and ketones (teacher version) and read these notes.

Each pair will require access to:

- pentan-3-one (**1A**)
- hexane (**1B**)
- butanal (**2B)** and (**4A**)
- ethyl ethanoate (**2A**)
- propanone (**3B**)
- pentan-3-one (**3A**)
- ethanoic acid (**4B**)
- sodium hydroxide solution [1.0 mol dm^{-3}]
- iodine solution [0.5 mol dm^{-3}] in potassium iodide solution [0.2 mol dm^{-3}]
- Fehling's solutions (freshly prepared from solutions A and B)
- Tollens' reagent (which should be freshly prepared from silver nitrate solution [0.1 mol dm^{-3}] in ammonia solution [2 mol dm^{-3}])
- Brady's reagent in phosphoric acid
- an electric kettle to make up a hot water-bath. Alternatively, use thermostatically controlled water-baths.
- clean test-tubes
- standard lab alcohol thermometers
- plastic pipettes
- 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
Pentan-3-one	 GHS02 (*flammable* **F**)  GHS07 (*moderate hazard* **MH**)	**In the eye:** flood eye with gently-running tap water for 10 min and consult a doctor. **Swallowed:** wash out mouth. Do not induce vomiting. Consult a doctor. **Spilt on skin or clothing:** remove contaminated clothing. Wash affected area and clothing with plenty of water. **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 fires:** allow fires in sinks, etc. to burn out. Fires at 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

Substance	Hazard	First aid
		amounts with cloth. Rinse well. For larger amounts open windows, cover with mineral absorbent (e.g., cat litter), scoop into bucket and add water.
Hexane	 GHS02 (*flammable* **F**)  GHS07 (*moderate hazard* **MH**)  GHS08 (*health hazard* **HH**)  GHS09 (*hazardous to the aquatic environment* **N**)	**In the eye:** flood eye with gently-running tap water for 10 min. See doctor. **Swallowed:** wash out the mouth. Do not induce vomiting. See doctor. **Spilt on skin or clothing:** remove contaminated clothing. Wash affected area and clothing with plenty of water. **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 fires:** allow fires in sinks, etc. to burn out. Fires at 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 cloth. Rinse well. For larger amounts open windows, cover with mineral absorbent (e.g., cat litter), scoop into bucket and add water.
Butanal	 GHS02 (*flammable* **F**)  GHS07 (*moderate hazard* **MH**)  GHS08 (*health hazard* **HH**)	**In the eye:** immediately rinse the eye with gently-running water for 10 min. See a doctor. **Vapour breathed in:** remove the casualty to fresh air. Keep them warm. See a doctor if breathing is difficult. **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. 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.
Ethyl ethanoate	 GHS02 (*flammable* **F**)	**In the eye:** immediately rinse the eye with gently-running water for 10 min. See a doctor. **Vapour breathed in:** remove the casualty to fresh air. Keep them warm. See a doctor if breathing is difficult.

Substance	Hazard	First aid
	 GHS07 (*moderate hazard* **MH**)	**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. 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.
Propanone	 GHS02 (*flammable* **F**)  GHS07 (*moderate hazard* **MH**)	**In the eye:** flood the eye with gently-running tap water for 10 min. See a doctor. **Vapour breathed in:** remove the casualty to fresh air. Keep them warm. See a doctor if breathing is difficult. **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. **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 propanone 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 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.
Ethanoic acid	 GHS02 (*flammable* **F**)  GHS05 (*corrosive* **C**)	**In the eye:** flood the eye with gently running tap water for 10 min. See a doctor. **Vapour breathed in:** remove to fresh air. Call a doctor if breathing is difficult. **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 doctor. **Spilt on the skin or clothing:** remove contaminated clothing. Then drench the skin with plenty of water. If a large area

Substance	Hazard	First aid
		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 large spills, and especially for (moderately) concentrated acid, cover with mineral absorbent (e.g., cat litter) and scoop into bucket. Neutralise with sodium carbonate. 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. 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 mol dm^{-3}). 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 mol dm^{-3}) to the remaining spill and leave for 1 h. Mop up and rinse with plenty of water.

Substance	Hazard	First aid
Fehling's A (copper(II) sulfate)	 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.
Fehling's B (potassium sodium tartrate)	 GHS05 (*corrosive* **C**)  GHS07 (*moderate hazard* **MH**)	**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. **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.
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.
Ammonia solution [2.0 mol dm^{-3}]	 GHS05 (*corrosive* **C**)	**In the eye:** flood the eye with gently-running tap water for at least 20 min (for alkalis). See a doctor. If it is necessary to go to hospital, continue washing the eye during the journey in an ambulance.

Substance	Hazard	First aid
	 GHS07 (*moderate hazard* **MH**)  GHS09 (*hazardous to the aquatic environment* **N**)	**Vapour breathed in:** remove the casualty to fresh air. Call a doctor if breathing is difficult. **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. 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.:** consider the need to evacuate the laboratory and open windows if large amounts are spilt 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. Wipe up small amounts with a damp cloth and rinse it well.
Brady's reagent (in phosphoric acid) – pre-prepared commercially available solution	 GHS02 (*flammable* **F**)  GHS07 (*moderate hazard* **MH**)	**In the eye:** rinse with water for several minutes. Consult a doctor **If swallowed:** rinse mouth out with water and consult a doctor. **In case of fire:** allow fires in sinks, etc. to burn out. Fires at top of test-tubes, beakers, etc. should be smothered with a damp cloth or heat-proof mat. **Spilt on the floor, bench, etc.:** wear eye protection and gloves. Rinse the area with water and wipe up, rinsing repeatedly. 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

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 experiment 3 in a well-ventilated area 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 pairs 2 (and 3 if needed) 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.

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.

Preparation of Fehling's solution

Fehling's A
7 g $CuSO_4 \cdot 5H_2O$ dissolved in 100 cm^3 distilled water.

Fehling's B
35 g of potassium tartrate and 10 g of sodium hydroxide in 100 cm^3 of distilled water

To prepare Fehling's solution mix equal quantities of solution A and solution B in a beaker. The resulting solution will be a deep blue colour.

Preparation of Tollens' reagent

Add 50 cm^3 of 0.1 mol dm^{-3} silver nitrate to a beaker and add 2.0 mol dm^{-3} ammonia solution drop wise. A brown precipitate of silver(I) oxide forms. More ammonia solution is added until a clear solution forms. Then 23 cm^3 of 0.8 mol dm^{-3} potassium hydroxide is added and again a precipitate will form. More ammonia solution is then added until the solution becomes clear.

Steps	Notes
1. Check that learners have access to all of the equipment and chemicals they need for this experiment.	
2. **Pair 1: pentan-3-one (1A) and hexane (1B)** – the test with Brady's reagent, 2,4-dinitrophenylhydrazine solution	*Make sure gloves are used in this experiment. Remind learners that the reagents used here are flammable.*
3. Approximately 1 cm^3 of Brady's reagent is placed in two separate test-tubes.	*It is sufficient to use a plastic pipette for this.*
4. Add the test substances 1A and 1B (0.5 cm^3) and gently agitate.	*An orange precipitate should quickly form with sample 1A.*
5. Once the test is complete, flush the contents of the test-tubes down the sink with running water.	*Be vigilant that learners do this without splashing the residues.*
6. **Pair 2: ethyl ethanoate (2A) and butanal (2B)** – Tollens' or Fehling's test	*Depending on their original plan, learners may use either Tollens' reagent or Fehling's solution here (this is equally true for pair 4). Here we detail the Tollens' test. The reagent should be freshly prepared shortly before the lesson.*
7. Approximately 1 cm^3 of freshly prepared Tollens' reagent is placed in two separate test-tubes.	*It is sufficient to use a plastic pipette for this. The test-tubes need to be clean and dry (or ideally new). Otherwise no silver mirror will form.*
8. Add the test substances 2A and 2B (0.5 cm^3) and gently agitate.	
9. Place the test-tubes in a water-bath at 50 °C.	*A water-bath will need to be ready at approx. 50 °C. Almost immediately, a silver mirror should form with sample B. If a grey or brown solution forms, the test-tube was probably not clean or the silver nitrate solution too old.*

10. **Pair 3: pentan-3-one (3A) and propanone (3B)** – the iodoform (tri-iodomethane) test

The product of this reaction has strong odour. It is advisable to do this experiment in a fume cupboard or a well-ventilated area of the lab.

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

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

13. Finally, 10 drops of sodium hydroxide solution are added to each tube. Swirl gently and wait 2 min.

If no reaction occurs in either test-tube, warm gently in a water-bath at 50 °C.

Sample 3B should produce a positive test. A yellow precipitate of tri-iodomethane should be produced. Learners may notice the characteristic 'medical' smell produced in the positive test. This is due to the presence of tri-iodomethane.

14. **Pair 4: butanol (4A) and ethanoic acid (4B)** – Tollens' or Fehling's test

As stated above, depending on their original plan, learners may use either Tollens' reagent or Fehling's solution here. Here we detail the Fehling's test. The reagent should be freshly prepared shortly before the lesson.

15. Approximately 1 cm^3 of freshly prepared Fehling's reagent is placed in two separate test-tubes.

16. Add the test substances 4A and 4B (0.5 cm^3) and gently agitate.

17. Place in a hot water-bath for approx. 5 min.

Sample 4A should gradually change colour from blue to red.

18. 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.

Clean-up

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: Redox pathways

Resources	• Worksheet E
Learning objectives	By the end of the lesson: • ***all*** learners should be able to correctly identify some of the redox reactions, and suggest suitable reagents and conditions for them • ***most*** learners should be able to correctly identify the majority of the redox reactions and suggest suitable reagents and conditions for them • ***some*** learners will be able to correctly identify all of the redox reactions and suggest suitable reagents and conditions for them.

Timings	Activity
5 min 5min	**Starter/Introduction** Discuss the meaning of redox reactions. Ask learners, in pairs, to try to recall the various definitions of redox: • addition or removal of oxygen (or hydrogen); ask learners if burning magnesium in air causes the magnesium to be reduced or oxidised. • OILRIG: **O**xidation **I**s **L**oss (of electrons), **R**eduction **I**s **G**ain (of electrons). • increase/decrease in oxidation state.
5 min	**Main lesson** Explain to the learners that most of the examples in which they have seen redox operating have been inorganic reactions. However, redox reactions are very important in organic chemistry. Ask learners if they know what happens to ethanol when it is left in a narrow-neck opened bottle (*you may like to cite the example of wine 'going off' due to aerial oxidation – if appropriate*). Go on to emphasise that the ethanol slowly oxidises to ethanoic acid and that ethanoic acid contains a higher percentage of oxygen in its molecule (when compared to ethanol). It might also be worth mentioning that this process is very slow and is usually accelerated by the presence of bacteria in the air which promote the conversion.
30 min	Hand out Worksheet E to pairs of learners. The objective of this exercise is to encourage learners to think more deeply about redox reactions and consider the effects of oxidising and reducing reagents on a range of organic compounds. It would be worth emphasising that redox reagents vary in their strength and this dictates their uses. For example, lithium aluminium hydride is a very powerful reducing agent whereas sodium borohydride is milder.
10 min	Once the learners have completed the Worksheet, they should peer-assess each other's work and check for errors.
10 min	**Plenary** Learners compare their suggested solutions to those given in the suggested answers.

Worksheets and answers

	Worksheet	Answers
For use in *Briefing lesson*:		
A: Carbonyl containing compounds	**20**	**25**
For use in *Planning lesson*:		
B: Experiment design	**21**	**26**
For use in *Lab lesson*:		
C: Experiment guidance	**22**	**—**
D: Further experiment guidance	**23**	**—**
For use in *Debriefing lesson*:		
E: Redox pathways	**24**	**28**

Worksheet A: Carbonyl containing compounds

Study the following organic compounds.

A: $CH_3CH_2CH_2COOH$

B: $CH_3CH_2COCH_3$

C: $CH_3(CH_2)_3CH_2CHO$

D: $CH_3CH_2OCH_2CH_3$

E: $ClOCCH_2CH_3$

F: CH_3COOCH_3

G: $HOCCH_2C_6H_5$

H: $CH_3CH_2CONH_2$

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

In your lab book:

- Draw each of the structures **A**–**H**.
- Identify the compounds which contain a carbonyl group.
- Name each of the carbonyl containing compounds and indicate which of the molecules are ketones and which are aldehydes.

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:** pentan-3-one and hexane
- **Pair 2:** butanal and ethyl ethanoate
- **Pair 3:** propanone and pentan-3-one
- **Pair 4:** butanal and ethanoic acid

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

Your task is to choose a suitable qualitative organic test from the list below to perform on each pair of liquids. You may use the following analytical tests **once only** each:

- Tollens' reagent test
- Fehling's reagent test
- 2,4-DNPH test
- Tri-iodomethane test (iodoform test)

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.

Here is an example:

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 0.5 cm^3 of the test substance to a clean test tube. Next add 1 cm^3 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.*
Safety and disposal: *The small quantities used in the experiment can safely be flushed 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: pentan-3-one and hexane	
Reagents	Brady's reagent (2,4-dinitrophenylhydrazine solution in phosphoric acid)
Equipment	Test-tubes, plastic pipettes
Safety	Gloves should be worn. Pentan-3-one, hexane and Brady's reagent are all flammable.
Procedure and conditions	Add the test substance (0.5 cm^3) to a solution of the Brady's reagent (1 cm^3) in a test tube.
Disposal	Flush the contents of the test-tubes down the sink with water.

Pair 2: butanal and ethyl ethanoate	
Reagents	Tollens' reagent*
Equipment	Test-tubes, plastic pipettes, warm water-bath
Safety	Tollens' reagent is corrosive and so gloves should be worn. Butanal and ethyl ethanoate are flammable and are also irritants.
Procedure and conditions	Add 0.5 cm^3 of the test substance to a clean test-tube containing freshly prepared Tollens' reagent (1 cm^3). The tubes are shaken gently and placed in a warm water-bath at 50 °C.
Disposal	As soon as the test is complete, flush the contents of the test-tubes down the sink with water.

Pair 3: propanone and pentan-3-one	
Reagents	Iodine solution and 1.0 mol dm^{-3} sodium hydroxide solution
Equipment	Test-tubes, plastic pipettes, water-bath at 50 °C (if needed)
Safety	Gloves should be worn, and this test should be performed in a fume cupboard. Sodium hydroxide is corrosive, and all of the chemicals are irritants. Propanone and butanone are also flammable.
Procedure and conditions	The test substance (0.5 cm^3) is added to a clean test-tube. Iodine solution (1 cm^3) is added, followed by 1 mol dm^{-3} sodium hydroxide solution (0.5 cm^3). The mixture is shaken gently (and warmed gently if necessary).
Disposal	The contents should be flushed down the sink with running water, preferably in the fume cupboard.

Pair 4: butanal and ethanoic acid	
Reagents	Fehling's reagent*
Equipment	Test-tubes, plastic pipettes, a hot water-bath
Safety	Fehling's reagent and ethanoic acid are corrosive. All of the substances are irritants. Butanal and ethanoic acid are flammable.
Procedure and conditions	Add 0.5 cm^3 of the test substance to a test-tube containing freshly prepared Fehling's solution (1 cm^3). Heat in a hot water-bath at 80 °C for 5 min.
Disposal	This reagent contains the heavy metal copper. Therefore, the contents of the test-tubes should be placed in the metals recovery bottle provided.

**Note that the tests used for pairs 2 and 4 may be interchanged.*

Worksheet E: Redox pathways

1. In the experimental lesson you used Tollens' and Fehling's reagents, performed the tri-iodomethane (iodoform) reaction and used 2,4-dinitrophenylhydrazine (Brady's reagent).

   Consider the use of each of these substances and write down in your lab books:

   a. Which of the reactions were redox reactions?

   b. For those reactions you marked as redox reactions, which experimental observations led you to believe this?

2. Consider the following organic reactions:

   A: pentan-3-one to pentan-3-ol

   B: butanol to butanoic acid

   C: butanoic acid to butan-1-ol

   D: butanal to butan-1-ol

   E: decan-1-ol to decanal

   F: propanol to propanoic acid

   For each reaction:

   a. Decide if the starting compound is being reduced or oxidised.

   b. Choose a reagent from the list opposite that would be suitable to carry out the reaction.

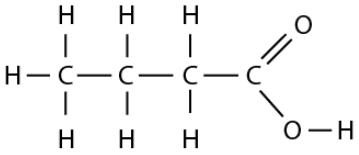
   c. Briefly describe the conditions necessary to carry out each reaction.

Reagents:
- lithium aluminium hydride
- acidified potassium dichromate solution
- sodium borohydride
- acidified potassium manganate (VII) solution

You are strongly advised to draw out each of the structures before you attempt this question

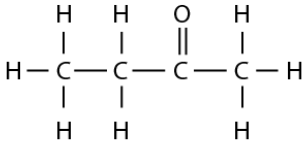
Worksheet A: Answers

A



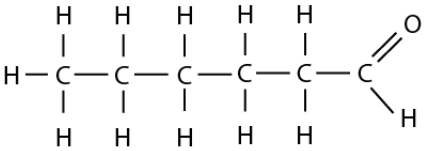
Butanoic acid (carboxylic acid)

B



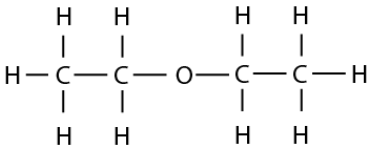
Butanone (ketone)

C



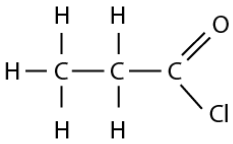
Hexanal (aldehyde)

D



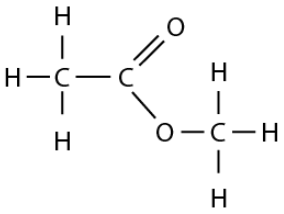
Ethoxyethane / diethyl ether (ether)

E



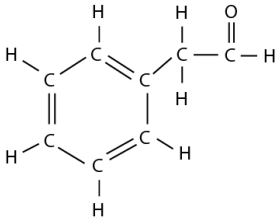
Propanoyl chloride (acid chloride)

F



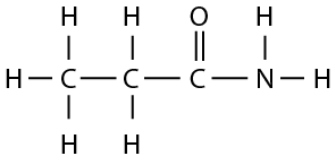
Methyl ethanoate (ester)

G



2-phenylethanal (aldehyde)

H



Propanamide (primary amide)

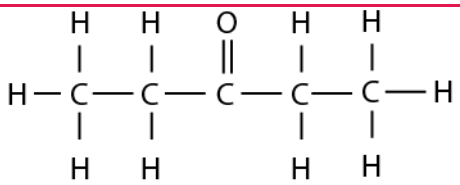
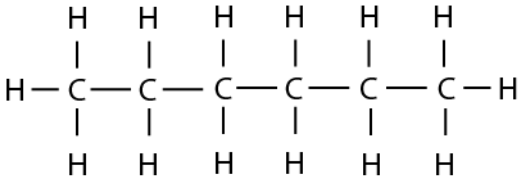
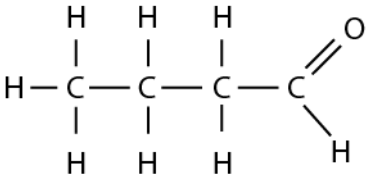
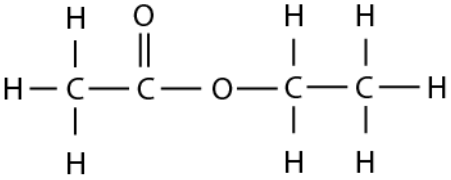
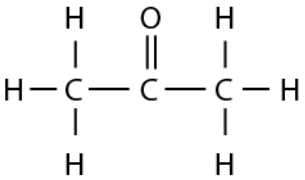
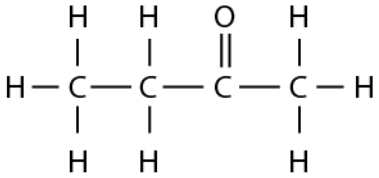
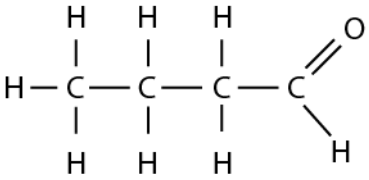
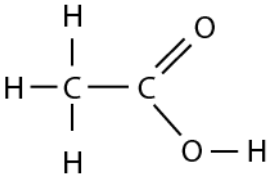
All of the compounds contain a carbonyl group except the ether, **D**.

Compounds **C** and **G** are aldehydes

Compound **B** is a ketone

Worksheet B: Answers

1.

Pair 1: pentan-3-one and hexane		
Pair 2: butanal and ethyl ethanoate		
Pair 3: propanone and butanone		
Pair 4: butanal and ethanoic acid		

2.

Pair 1: pentan-3-one and hexane
Functional group(s) present: • *ketone = RCOR'* • *Hexane does not contain a functional group.*
Name of the test required: *Use 2,4-dinitrophenylhydrazine solution (Brady's reagent).*
Explanation of why test is required: *2,4-Dinitrophenylhydrazine will react with the carbonyl group in the ketone to form an orange-coloured precipitate. There will be no reaction with hexane since it does not contain a carbonyl group.*
Description of the test: *Add the test substance (0.5 cm^3) to a solution of the 2,4-dinitrophenylhydrazine solution in a test-tube. A positive test should result in the immediate formation of an orange precipitate.*
Safety and disposal: *Flush the contents of the test-tubes down the sink with running water.*

Worksheet B: Answers, continued

Pair 2: butanal and ethyl ethanoate
Functional group(s) present: • *aldehyde = RCOH* • *ester = RCOOR'*
Name of the test required: *Use Tollens' reagent, which will react with the aldehyde butanal to form a silver mirror. The ester will not react. (Fehling's reagent could also be used here.)*
Explanation of why test is required: *The aldehyde is able to reduce the silver ions to elemental silver in the Tollens' reagent. The ester cannot do this.*
Description of the test: *Add 0.5 cm^3 of the test substance to a clean test-tube containing freshly prepared Tollens' reagent (1 cm^3). The tubes are shaken gently and placed in a warm water-bath. A silver mirror is formed with the aldehyde but not with the ester.*
Safety and disposal: *Learners may understandably suggest that the silver residues should be stored in a heavy metals waste bottle, but in this case, the contents of the tubes should be flushed down the sink to prevent the possibility of explosive by-products being formed.*

Pair 3: propanone and pentan-3-one
Functional group(s) present: *Both molecules are ketones = RCOR'*
Name of the test required: *The tri-iodomethane test (iodoform test).*
Explanation of why test is required: *Only methyl ketones react to produce a yellow precipitate of tri-iodomethane. Therefore, only propanone will produce a positive test.*
Description of the test: *The test substance (0.5 cm^3) is added to a clean test-tube. Iodine solution (1 cm^3) is added, followed by 1 mol dm^{-3} sodium hydroxide solution (0.5 cm^3). The mixture is shaken gently and warmed. A pale-yellow precipitate should only form in the case of propanone since it is a methyl ketone.*
Safety and disposal: *The contents of the tubes should be allowed to evaporate at the back of the fume cupboard and the remainder flushed down the sink with a plentiful supply of running water.*

Pair 4: butanal and ethanoic acid
Functional group(s) present: *Aldehyde = RCOH and carboxylic acid RCOOH.*
Name of the test required: *Use the Fehling's test (or Tollens' test).*
Explanation of why test is required: *The Fehling's test will be positive for the aldehyde but will not work with the carboxylic acid.*
Description of the test: *Add 0.5 cm^3 of the test substance to a test-tube containing freshly prepared Fehling's solution (1 cm^3). Heat in a hot water-bath for 5 min. The aldehyde should react to produce a brick-red precipitate. The other test-tube should remain blue.*
Safety and disposal: *Copper residues should be stored in a heavy metals waste bottle.*

Worksheet E: Answers

1.

a. The use of Tollens', Fehling's and the tri-iodomethane (iodoform) involve redox reactions.

b. **Tollens' reagent**: the presence of a silver mirror indicated the presence of elemental silver in oxidation state = 0. Silver in silver nitrate, $AgNO_3$, is in oxidation state +1. Therefore, this is a redox reaction.

Fehling's reagent: This reaction is accompanied by a change in colour from a blue solution of copper(II) ions to a precipitate of red/orange copper(I) ions.

The tri-iodomethane (iodoform) reaction: elemental iodine, with oxidation state = 0, is used in this reaction. In a positive test, the dark brown colour of the elemental iodine disappears as it is reduced to iodide ions in the -1 oxidation state. [Note that iodide ions in the sodium iodide are colourless, but a pale-yellow precipitate of iodoform = CH_3I is observed in the test tube.]

2.

Pentan-3-one to pentan-3-ol

This is a reduction. Either lithium tetrahydridoaluminate (lithium aluminium hydride), $LiAlH_4$, or sodium tetrahydridoborate (sodium borohydride), $NaBH_4$, may be used. However, it is advisable to use the latter reagent since it is safer and milder. If $NaBH_4$ is used, the reaction may be performed in aqueous alkaline conditions or as a solution in ethanol.

Butanol to butanoic acid

This is an oxidation. An excess of **acidified** potassium dichromate solution should be used under reflux conditions.

Butanoic acid to butan-1-ol

This is a reduction. Powerfully reducing conditions are needed to effect this transformation and therefore lithium tetrahydridoaluminate (lithium aluminium hydride), $LiAlH_4$, is used. Since the reagent is so powerfully reducing, the reaction occurs at room temperature in dry ethoxyethane (diethyl ether).

Worksheet E: Answers, continued

Butanal to butan-1-ol

This is a reduction. Mildly reducing conditions are sufficient for this conversion using sodium tetrahydridoborate (sodium borohydride), $NaBH_4$. The conditions may be aqueous alkaline or as a solution in ethanol.

Decan-1-ol to decanal

This is an oxidation. A stoichiometric quantity of acidified potassium dichromate solution (or a slight excess of the alcohol) should be used. As the aldehyde is produced it must be distilled off to prevent further oxidation (to the carboxylic acid).

Propanol to propanoic acid

This is an oxidation. An excess of ***acidified*** *potassium dichromate solution should be used under reflux conditions. An alternative reagent to use acidified potassium manganate(VII) solution (but this is not a clean reaction).*

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