Qualitative tests for organic functional groups

A video featuring this experiment is available at <u>https://rsc.li/38KQFpE</u>, along with teacher notes and worksheets for learners.

Equipment (per group)

- 22 x test tubes and spares
- 6 x bungs for test tubes (minimum)
- 1 x test tube rack (minimum)
- 12 x dropper pipettes

Safety equipment: safety spectacles

Preparation

Solutions to be either made up in small dropper bottles enough for one set per group. Alternatively make up in larger bottles for students to share between groups.

Each group will need the following chemicals:

- sample A cyclohexene
- sample B ethanol or propan-1-ol
- sample C ethanal or propanal
- sample D propanone
- sample E 1-bromobutane (could also use either chloro or iodobutane)
- sample F dilute ethanoic acid
- bromine water, 0.2 mol dm⁻³
- sodium hydroxide solution, 0.4 mol dm⁻³

2 x well plates or spotting tiles

2 x beakers, 400 cm³ (for water baths)

1 x kettle to boil water for water bath

- nitric(v) acid solution, 0.4 mol dm⁻³
- silver nitrate solution, 0.1 mol dm⁻³
- potassium dichromate solution 0.1 mol dm⁻³ (2 cm³)
- sulfuric acid solution, 2.0 mol dm-3 (10 cm³)
- sodium or potassium carbonate solution, 0.4 mol dm⁻³
- Brady's reagent (see preparation information below)
- chemicals to prepare Tollens' reagent: silver nitrate solution 0.1 mol dm⁻³ and ammonia solution, 1.00 mol dm⁻³

Equipment set-up and tips

Learners carrying out this practical are encouraged to plan the order they do the tests and to start with the tests using the least hazardous reagents. Once they have identified a sample they will not need to continue to test that one. Set-up for each stage is shown below and the time stamp for the relevant section of the <u>video</u> is given for easy reference.



1. **Test for carboxylic acid** by adding a few drops of sodium hydrogen carbonate solution to each sample in a labelled test tube, see 02:04.



2. Test for an unsaturated hydrocarbon by adding 1 cm³ of bromine water to each sample, see 03:35.



3. **Test for haloalkanes**: 10 drops of each sample is combined with 1 cm³ sodium hydroxide solution and 2 cm³ of ethanol in one set of labelled boiling tubes; 2.5 cm³ silver nitrate solution is put into a second set, see 05:26. A few drops of dilute nitric acid are added to the sample mixtures before the silver nitrate is added.

Use two water baths, with half of the water from a freshly boiled kettle and the other half from a cold tap.



4. **Test for alcohols** with acidified potassium dichromate solution, conducting this test in microscale means only a small amount of the reagent is used, see 07:40.



5. **Test for carbonyl groups** with Brady's reagent – a solution of dinitrophenylhydrazine (2,4-DNPH), also microscale, see 09:49.



A silver mirror will from in the presence of an aldehyde. Dispose of the reaction mixture straight away down a foul-water drain.

Expected	results
Lycorda	resares

Test for	carboxylic acid	unsaturated hydrocarbon	haloalkanes	alcohols	carbonyl groups	aldehydes	
Test used	metal carbonate	bromine water	silver nitrate solution	acidified potassium dichromate	2,4-DNPH	Tollens' reagent	Functional group present
A	no change	colour change: orange to colourless	no change	colour change: SLOW	no change		alkene
В	no change	no change	no change	colour change: orange to green	no change		alcohol
с	no change	colour change: orange to colourless	no change	colour change: orange to green	yellow/ orange precipitate	silver mirror	aldehyde
D	no change	no change	no change	no change	yellow/ orange precipitate		ketone
E	no change	no change	cream precipitate				haloalkane containing bromine
F	effervescence						carboxylic acid

Safety

Read our standard health & safety guidance and carry out a risk assessment before running any live practical.

Refer to SSERC/CLEAPSS Hazcards and recipe sheets.

Hazard classification may vary depending on supplier.

Chemical supplied for the practical	Preparation	
cyclohexene		

C₆H₁₀ (l)



DANGER Highly flammable liquid and vapour May be fatal if swallowed and enters airways Toxic/ very toxic to aquatic life with long lasting effect Harmful if swallowed

ethanol C₃H₅O (l)



DANGER Highly flammable *or* ethanol (IDA – contains 95% ethanol)



DANGER Highly flammable Harmful (ingestion) May cause damage to organs

propan-1-ol C3H8O (l)



DANGER Highly flammable liquid and vapour May cause drowsiness or dizziness Causes serious eye damage

ethanal C₂H₄O (l)



DANGER Extremely flammable liquid and vapour Causes serious eye irritation May cause respiratory irritation Suspected of causing genetic effects May cause cancer

TECHNICIAN NOTES

Chemical supplied for the practical

Preparation

propanal C₃H₆O (l)



Highly flammable liquid and vapour Causes skin irritation Causes serious eye irritation May cause respiratory irritation

propanone C₃H₆O (l)



DANGER

Highly flammable liquid and vapour Causes serious eye irritation May cause drowsiness or dizziness Repeated exposure may cause skin dryness and cracking

1-bromobutane C₄H₅Br (l)



DANGER

Highly flammable liquid and vapour One or more of the following may also apply: Causes skin irritation Causes serious eye irritation May cause respiratory irritation Toxic (or harmful) to aquatic life with long lasting effects

1-iodobutane C, H9, (l)



WARNING

Flammable liquid and vapour Harmful if swallowed, in contact with skin and if inhaled Causes skin and serious eye irritation may cause respiratory irritation

1-chlorobutane C₄H_qCl (l)



DANGER Highly flammable liquid and vapour

TECHNICIAN NOTES

Chemical supplied for the practical	Preparation
ethanoic acid solution	ethanoic acid concentrated
0.40 mol dm ⁻³	$C_2H_4O_2(l)$
$C_2H_4O_2$ (aq)	MW= 60.05 g mol ⁻¹
Currently not classified as hazardous	
	DANGER
	Flammable vapour and liquid
	Causes severe skin burns and eye damage
bromine water	bromine liquid
0.20 mol dm ⁻³	Br ₂ (l)
Br ₂ (aq)	MW= 160.00 g mol ⁻¹
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DANGER	DANGER
Corrosive (eyes)	Causes severe skin burns and eye damage
Irritant (skin)	Very toxic to aquatic organisms
	Inhalation of bromine vapour acts as a respiratory
	irritant
	(See further notes below)
sodium hydroxide solution	sodium hydroxide solid
0.40 mol dm ⁻³	NaOH (s)
NaOH (aq)	MW= 40.00 g mol ⁻¹
WARNING	DANGER
Irritant (skin, eyes)	Causes severe skin burns and eye damage
nitric(v) acid solution	nitric(v) acid concentrated solution
0.4 mol dm⁻³	HNO ₃ (aq)
HNO ₃ (aq)	MW= 63.01 g mol ⁻¹
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WARNING	DANGER
Irritant (skin, eyes)	May intensify fire; oxidiser
	Causes severe skin burns and eye damage
	Corrosive to the respiratory tract
silver nitrate solution, 0.10 mol dm ⁻³	silver nitrate solid
AgNO ₃ (aq)	AgNO ₃ (s)
\wedge	MW= 169.87 g mol ⁻¹
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WARNING	
Irritant (skin, eyes)	DANGER
	May intensify fire; oxidiser
	Causes severe skin burns and eye damage
	Very toxic to aquatic life with long lasting effects
	Note: Some suppliers may also indicate harmful if

swallowed

Chemical supplied for the practical	Preparation
potassium dichromate(vı) solution	potassium dichromate(vı) solid
0.10 mol dm ⁻³ in 1.40 mol dm ⁻³ sulfuric acid	K2Cr2O7 (s)
$K_2 Cr_2 O_7 (aq)$	MW= 294.19 g mol ⁻¹
DANGER	DANGER
Corrosive (skin, eyes)	May intensify fire; oxidiser
Harmful (ingestion)	Toxic if swallowed
Irritant (respiratory)	Harmful in contact with skin
Sensitiser (skin, respiratory)	Causes severe skin burns and eye damage
Serious Health Hazard (RE)	May cause an allergic skin reaction
Serious Health Hazard (CMR)	Fatal if inhaled
	May cause allergy or asthma symptoms or breathing
	difficulties if inhaled
	May cause genetic defects
	May cause cancer
	May damage fertility. May damage the unborn child
	Causes damage to organs through prolonged or
	repeated exposure
	Very toxic to aquatic life with long lasting effects
	(See further notes below)
sulfuric(vı) acid	sulfuric(vı) acid concentrated
1.4 mol dm ⁻³ (to make potassium dichromate solution)	H2SO4 (l)
H ₂ SO ₄ (aq)	MW= 98.07 g mol ⁻¹
	E.
WARNING	DANGER
Irritant (skin, eyes)	Cause severe skin burns and eye damage
sodium or potassium carbonate	sodium carbonate solid
0.4 mol dm ⁻³	Na2CO3 (s)
Na2CO3 (aq)	MW= 105.99 g mol ⁻¹
K ₂ CO ₃ (aq)	∧
Currently not classified as hazardous	
	\checkmark
	WARNING
	Causes serious eye irritation
	or
	potassium carbonate solid
	$K_2 CO_3 (s)$
	MW= 138.21 g mol ⁻¹
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WARNING Causes serious eye irritation

TECHNICIAN NOTES

Chemical supplied for the practical

Brady's reagent (ready-made solution)

Causes severe skin burns and eye damage

2,4-DNPH in phosphoric acid

Flammable liquid and vapour

May be corrosive to metals

ammonia (ammonium hydroxide) solution 1.00 mol dm⁻³ NH₂ (aq)



DANGER

WARNING Irritant (skin, eyes)

Preparation

ammonia solution "880", 35% w/w solution $\rm NH_{_3}\,(aq)$ MW= 17.01 g mol^1



DANGER

Causes severe skin burns and eye damage May cause respiratory irritation Very toxic to aquatic life (See further notes below)

2,4-dinitrophenylhydrazine solid C₀H₀N₄O₄ (s) MW= 198.14 g mol⁻¹



DANGER Flammable solid Harmful if swallowed

Phosphoric(V) acid liquid H₃PO (l) MW = 97.99 g mol⁻¹



DANGER Causes severe skin burns and eye damage

Ethanol liquid $C_2H_6O(l)$ MW = 46.07 g mol⁻¹



DANGER Highly flammable (See further notes below)

Wear eye protection.

Avoid skin contact and wear chemical resistant gloves if wounds or skin conditions exist.

Avoid breathing chemicals and keep bottles closed and keep bungs on test tubes as much as possible. Dispose of the reaction products after each step if possible.

Silver nitrate solution stains clothing and surfaces.

Bromine is acutely toxic by inhalation. Take great care when using bromine and wear splash proof goggles or a face shield and gloves and work in a fume cupboard. Always make sure there is someone in the room with you in case of emergency. If using a bromine ampoule, follow guidance from CLEAPSS or SSERC on how to open it. It's best to prepare quantity required only or the quantity that you might need over two weeks as bromine will be lost by diffusion, the solution will look paler and the concentration will decrease. However, this is not always possible if you use a bromine ampoule and you might want to prepare a higher concentration (eg 0.3%) and dilute the necessary volume to 0.1%. The remaining 0.3% solution can be tried and diluted accordingly at a later date. Any bromine water that has not been used should be decanted into a screw-top glass bottle for storage.

Potassium dichromate: inhalation of dichromate(VI) salts is a remote possibility if significant amounts of dust become airborne. Use correct transfer techniques. Do not allow small volumes of solution to dry out on glassware/lab surfaces; wipe up immediately with a damp cloth/paper towel. Avoid raising dust. Wear splash-proof goggles and take care to avoid skin contact.

To measure out the solid: use a balance placed in a fume cupboard that is not switched on. Have sash partially down. Wipe down the work area with a damp paper towel.

Ammonia, concentrated: wear splash-proof goggles. Protect the face when opening bottles of concentrated ammonia (pressure may have built up and the bottle might have changed shape and become unstable), or when transferring or dispensing large volumes. Use a fume cupboard. Do not inhale vapour. Take particular care to avoid skin contact.

Brady's reagent: make only the minimum quantity as and when required and then promptly dispose of any unused reagent by diluting the content in a bucket of water and pouring down a foul-water drain with more water; do not store the solution. Brady's reagent can be purchased ready made from chemical suppliers. If using an existing stock of solid 2,4-DNPH, check that it has been stored in an outer container of water. Do not open a container that may contain dried out 2,4, DNPH.

To prepare 25 cm³ of solution of dinitrophenylhydrazine (2,4-DNPH)

Note that methods using concentrated sulfuric acid and methanol are no longer recommended. Wear splashproof goggles and chemical resistant gloves. Protect the bench to avoid staining.

- 1. Add 0.5 g of 2,4-DNPH to about 12–13 cm³ of 85% phosphoric(v) acid. Do not use old phosphoric acid for the preparation of Brady's reagent. Phosphoric acid undergoes polymerisation during long-term storage leading to the build-up of solid at the bottom of the bottle.
- 2. Stir until the 2,4-DNPH has completely dissolved in the acid before moving on to Step 3. Dissolving may take 10–15 minutes.
- 3. Once the 2,4-DNPH has dissolved, make up to a total volume of 25 cm³ with ethanol (or IDA). Stir to mix.

Disposal

Due to the very small scale of each reaction, these small quantities can be put down a foul-water drain with further dilution. Students should empty their test tubes from parts 1–3 into a labelled waste container after each step if possible and before they move on to parts 4 and 5. The spotting tiles used for parts 4 and 5 can be rinsed due to the small amount of chemicals used. Therefore, students shouldn't have any waste from previous tests when they start the Tollens' test, to avoid anyone disposing of the Tollens' mixture in the waste container by mistake. Dispose of the Tollens' reagent straight after use.