# Edexcel

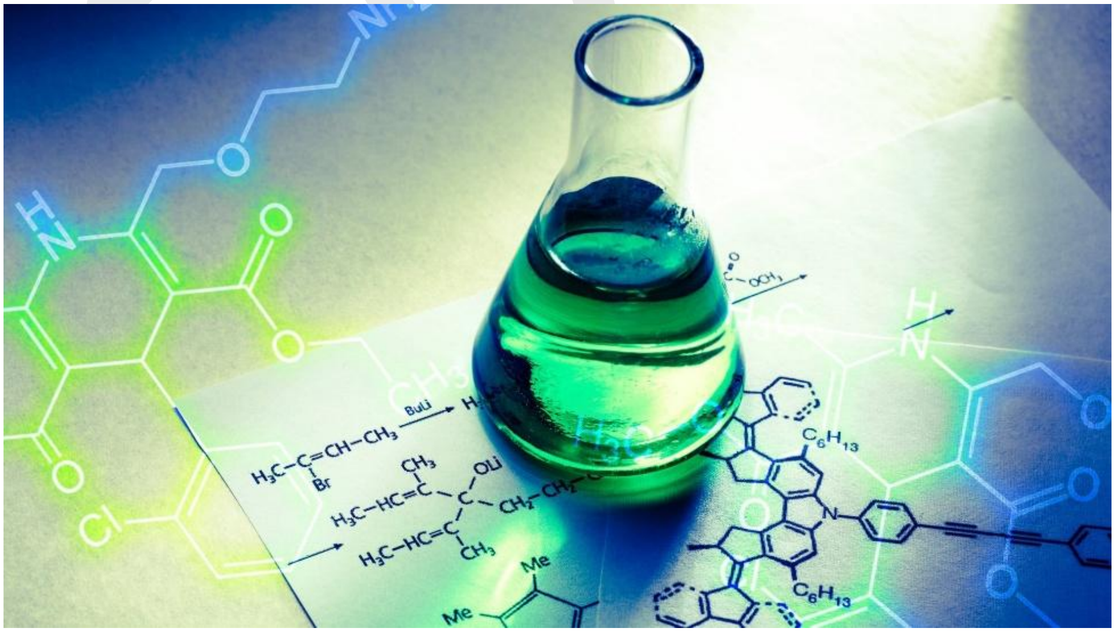
# A - Level

# Chemistry

CODE: (WCH11)

## Topic 20

# Organic synthesis



# 20A 1 Organic analysis

## TRADITIONAL METHODS

The traditional method of analysis is to find the empirical formula, followed by the molecular formula and then the functional groups present.

## FINDING THE EMPIRICAL AND MOLECULAR FORMULAE

You learned the method for finding the empirical formula of a compound in Topic 1 (Book 1: IAS).

To find the empirical formula from the percentage composition by mass of the elements you must first divide the percentage composition of each element by its relative atomic mass.

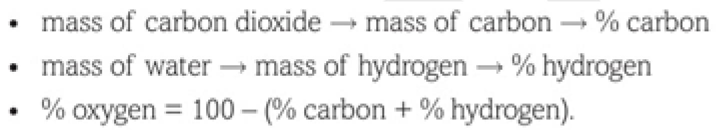
You then divide the values obtained by the smallest value. The numbers you obtain should give you an obvious whole number ratio.

## COMBUSTION ANALYSIS

We can use combustion analysis to find the percentage composition of a compound.

A known mass of the compound is burned in excess dry oxygen. The masses of carbon dioxide and water produced are measured.

For a compound that contains carbon, hydrogen and oxygen only we can then work out the following:



- mass of carbon dioxide → mass of carbon → % carbon
- mass of water → mass of hydrogen → % hydrogen
- % oxygen = 100 – (% carbon + % hydrogen).

## CHARACTERISTIC REACTIONS OF FUNCTIONAL GROUPS

To determine the structural formula from the molecular formula we need to identify the functional groups in the compound.

Traditionally, this is done by performing reactions in test tubes, using test reagents such as bromine water or 2,4-dinitrophenylhydrazine.

A reminder of these tests is given in table A.

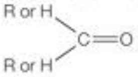
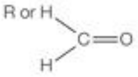
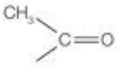
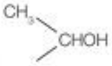
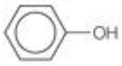
| FUNCTIONAL GROUP | TEST | OBSERVATION | NOTES |
|---|---|---|---|
| Alkene (C=C) | add $Br_2(aq)$ | bromine decolourises immediately | aldehydes and ketones decolourise $Br_2(aq)$, but very slowly<br>phenols decolourise $Br_2(aq)$ immediately and also produce a white precipitate |
| Halogenoalkanes (R–X), where X = Cl, Br or I | warm with NaOH(aq) and then add dilute $HNO_3$, followed by a few drops of $AgNO_3(aq)$ | R–Cl produces a white precipitate<br>R–Br produces a cream precipitate<br>R–I produces a yellow precipitate | the precipitate is insoluble in dilute $NH_3(aq)$<br>the precipitate is insoluble in dilute $NH_3(aq)$, but soluble in concentrated $NH_3(aq)$<br>the precipitate is insoluble in both dilute $NH_3(aq)$ and concentrated $NH_3(aq)$ |
| Hydroxy group (–OH) | add solid $PCl_5$ | misty fumes | both alcohols and carboxylic acids, but not phenols, produce misty fumes |
| Primary alcohol ($RCH_2OH$) | add acidified $K_2Cr_2O_7$ solution and warm | solution turns from orange to green | the organic product of this reaction produces a silver mirror with Tollens' reagent |
| Secondary alcohol ($R_2CHOH$) | add acidified $K_2Cr_2O_7$ solution and warm | solution turns from orange to green | the organic product does not produce a silver mirror with Tollens' reagent |
| Carbonyl group  | add 2,4-dinitrophenylhydrazine | orange precipitate | both aldehydes and ketones produce an orange precipitate |
| Aldehydes  | add Tollens' reagent and warm | silver mirror forms | Fehling's or Benedict's solutions can also be used<br>ketones do not react with Tollens' reagent, Fehling's solution or Benedict's solution |
|  or  | add an alkaline solution of iodine and warm | yellow precipitate forms | this is known as the triiodomethane (or iodoform) test |
| Carboxylic acids (R–COOH) | add $NaHCO_3(aq)$ or $Na_2CO_3(aq)$ and warm if necessary | bubbles of gas | |
| Phenol  | add bromine water | bromine immediately decolourises and a white precipitate forms | phenylamine produces the same observation<br>however, phenol is soluble in NaOH(aq) but insoluble in dilute HCl; phenylamine is insoluble in NaOH(aq) but soluble in dilute HCl |

**table A** Tests for functional groups.

### MODERN ANALYTIC TECHNIQUES

Chemists use a combination of techniques to identify organic compounds and determine their structures, including:

- mass spectra
- infrared spectra
- NMR spectra (both $^{13}C$ and proton).

Mass spectrometry determines the relative molecular mass of a compound, while infrared spectroscopy identifies functional groups and features in organic compounds. $^{13}C$ NMR spectroscopy identifies different carbon atom environments in compounds, such as $CH_3CH_2OH$ and $CH_3CH_2COOCH(CH_3)_2$, with five peaks for ethanol and six peaks for 1-methylethyl propanoate. Proton NMR spectroscopy identifies hydrogen atom groups in compounds.

## 20A 2 Organic synthesis

### WHAT IS ORGANIC SYNTHESIS?

Synthesis is the process of creating something new from existing compounds, often using a familiar compound to create an unfamiliar one. This book focuses on a two-step synthesis, requiring multiple steps. Planning a reaction scheme is challenging and requires knowledge of various chemical reactions. An example is converting bromoethane to ethyl propanoate, which requires considering reactions of bromoethane and the reactions resulting in ethyl propanoate.

You could consider the question as a puzzle in which you need to find the identity of X in this sequence:
bromoethane→ X → ethyl propanoate

### PREPARING FOR PLANNING A SYNTHESIS

One thing that will help you learn how to plan a synthesis is to put together an outline of all the reactions you need to know. You could leave out reactions that are less useful in synthesis, such as combustion, polymerisation and chemical tests. You could try to produce a diagram (like a spider diagram) that would include all the reactions, but that would probably be too complicated to easily use. It might be better to produce a series of smaller ones. For example, fig A shows reactions based on alcohols.

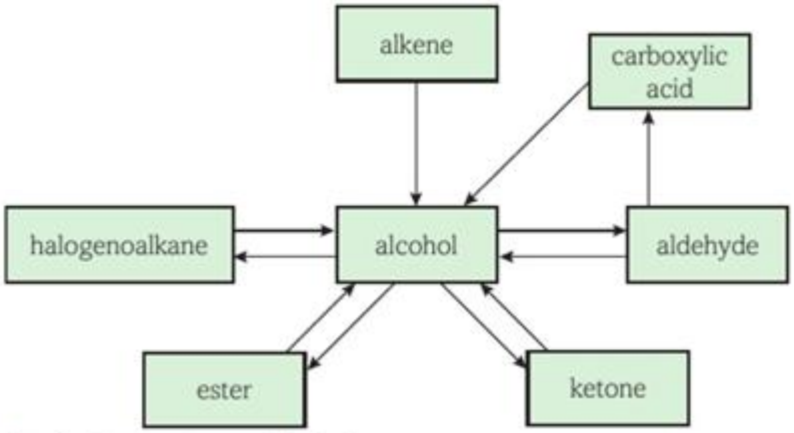


**fig A** Reactions of alcohols.

## EXTENDING A CARBON CHAIN

It is possible that one of the steps in your synthesis will involve extending an existing carbon chain by one or more carbon atoms. You have already learned three ways of doing this:

1 Reacting a halogenoalkane with a cyanide ion forms a nitrile with one more carbon atom than the halogenoalkane.

2 The addition of hydrogen cyanide to a carbonyl compound.

3 The alkylation of benzene, which introduces an alkyl group into a benzene ring.
A different way, which is more versatile, involves the use of Grignard reagents.

## GRIGNARD REAGENTS

Victor Grignard, a French chemist, developed a new method for synthesizing organic compounds using Grignard reagents, which are organometallic compounds containing magnesium, created by heating halogenoalkanes with magnesium in dry ether solvent.
A general equation for the formation of a Grignard reagent is:

$$R\text{—}Br + Mg \rightarrow R\text{—}Mg\text{—}Br$$

Grignard reagents react with water, so they are both made and used in a solvent of dry ether.

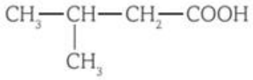
## GRIGNARD REACTIONS

Once a Grignard reagent has been made it can then be converted into a range of organic compounds, depending on the reagent used. In this book we will consider only two of these reactions: with carbon dioxide and with carbonyl compounds. They can be summarised as follows.

| | | | |
|---|---|---|---|
| 1 | with carbon dioxide | $RMgBr \rightarrow RCOOH$ | carboxylic acid |
| 2a | with methanal | $RMgBr \rightarrow RCH_2OH$ | primary alcohol |
| 2b | with an aldehyde R′CHO | $RMgBr \rightarrow RR'CHOH$ | secondary alcohol |
| 2c | with a ketone R′COR″ | $RMgBr \rightarrow RR'R''COH$ | tertiary alcohol |

## EXAMPLES OF GRIGNARD REACTIONS

### FORMATION OF 3-METHYLBUTANOIC ACID



$$CH_3\text{—}\underset{\displaystyle CH_3}{\underset{|}{CH}}\text{—}CH_2\text{—}COOH$$

This is an example of reaction 1, described above. The COOH part comes from carbon dioxide, so the starting compound must supply the $(CH_3)_2CHCH_2$ part, which means using $(CH_3)_2CHCH_2Br$, i.e. 1-bromo-2-methylpropane. The equations for the reactions that occur in this example are:

Step 1: formation of the Grignard reagent

$$(CH_3)_2CHCH_2Br + Mg \rightarrow (CH_3)_2CHCH_2MgBr$$

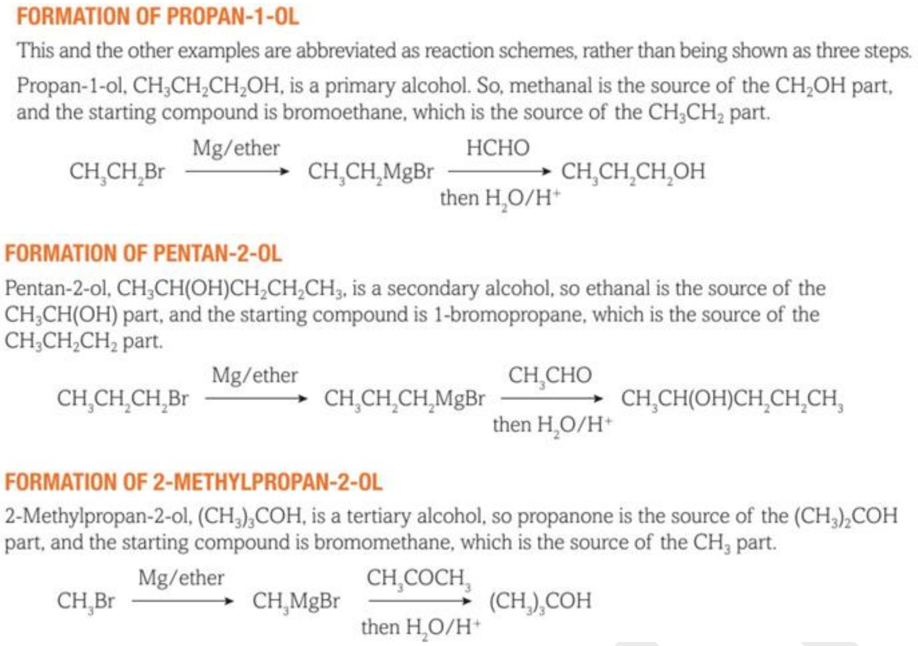
Step 2: reaction with chosen reagent

$$(CH_3)_2CHCH_2MgBr + CO_2 \rightarrow (CH_3)_2CHCH_2COOMgBr$$

Step 3: hydrolysis using a dilute acid

$$(CH_3)_2CHCH_2COOMgBr + H_2O \rightarrow (CH_3)_2CHCH_2COOH + Mg(OH)Br$$

The inorganic product of Step 3 will react with the dilute acid. You do not need to know details of the mechanisms.



### FORMATION OF PROPAN-1-OL

This and the other examples are abbreviated as reaction schemes, rather than being shown as three steps.

Propan-1-ol, $CH_3CH_2CH_2OH$, is a primary alcohol. So, methanal is the source of the $CH_2OH$ part, and the starting compound is bromoethane, which is the source of the $CH_3CH_2$ part.

$$CH_3CH_2Br \xrightarrow{Mg/ether} CH_3CH_2MgBr \xrightarrow[\text{then } H_2O/H^+]{HCHO} CH_3CH_2CH_2OH$$

### FORMATION OF PENTAN-2-OL

Pentan-2-ol, $CH_3CH(OH)CH_2CH_2CH_3$, is a secondary alcohol, so ethanal is the source of the $CH_3CH(OH)$ part, and the starting compound is 1-bromopropane, which is the source of the $CH_3CH_2CH_2$ part.

$$CH_3CH_2CH_2Br \xrightarrow{Mg/ether} CH_3CH_2CH_2MgBr \xrightarrow[\text{then } H_2O/H^+]{CH_3CHO} CH_3CH(OH)CH_2CH_2CH_3$$

### FORMATION OF 2-METHYLPROPAN-2-OL

2-Methylpropan-2-ol, $(CH_3)_3COH$, is a tertiary alcohol, so propanone is the source of the $(CH_3)_2COH$ part, and the starting compound is bromomethane, which is the source of the $CH_3$ part.

$$CH_3Br \xrightarrow{Mg/ether} CH_3MgBr \xrightarrow[\text{then } H_2O/H^+]{CH_3COCH_3} (CH_3)_3COH$$

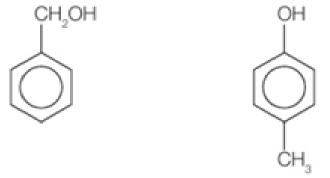
## PREDICTING THE CHEMICAL PROPERTIES OF COMPOUNDS

As we have seen in Section 20A.1, chemical reagents can be used to show which functional groups are present in a compound.

Functional groups can influence the behaviour of compounds that contain them. For example:

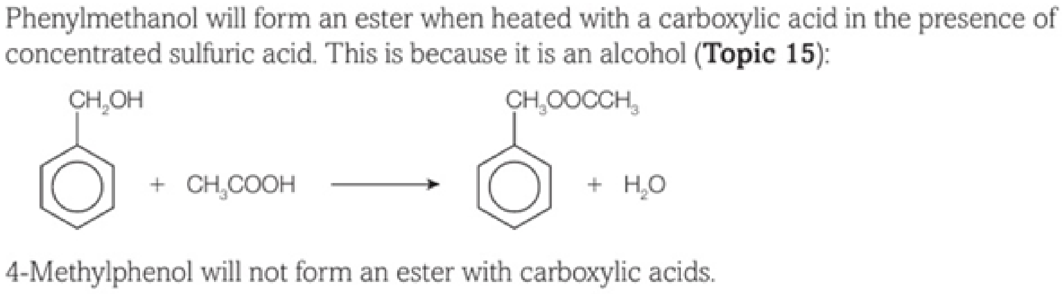
- some compounds will act as nucleophiles and others as electrophiles
- some will be susceptible to addition reactions and others to substitution reactions
- some will be easily oxidised and others are more easily reduced.

We can show this by looking at two compounds with the molecular formula C-H2O: phenylmethanol and 4-methylphenol (see fig C).



phenylmethanol 4-methylphenol

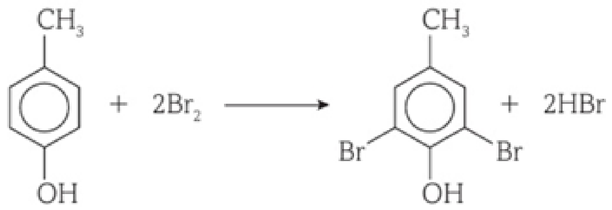
▲ **fig C**



Phenylmethanol will form an ester when heated with a carboxylic acid in the presence of concentrated sulfuric acid. This is because it is an alcohol (**Topic 15**):

4-Methylphenol will not form an ester with carboxylic acids.

4-Methylphenol will decolourise bromine water:



However, phenylmethanol does not react with bromine water.

## MULTI-STEP SYNTHESES

You may be asked to plan to a reaction scheme that shows all the intermediate compounds as well as the reagents and conditions required for each step. There are several ways you can approach this:

- by checking if the carbon chain length has increased or decreased
- by looking at the final product and 'working back' to the starting compound
- by looking at the starting compound and thinking of the types of reactions it can undergo.

Fig D shows how working back can be used to plan the synthesis of one compound from another. In this case the starting compound is bromoethane and the final compound is ethanoic acid:

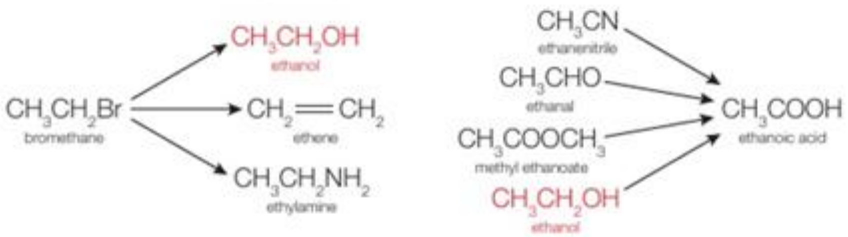


**fig D**

You can plan the reaction scheme using the following stages.

• Begin by writing the formulae of the compounds that can be readily converted into ethanoic acid. These include ethanenitrile, ethanol, methyl ethanoate and ethanol.

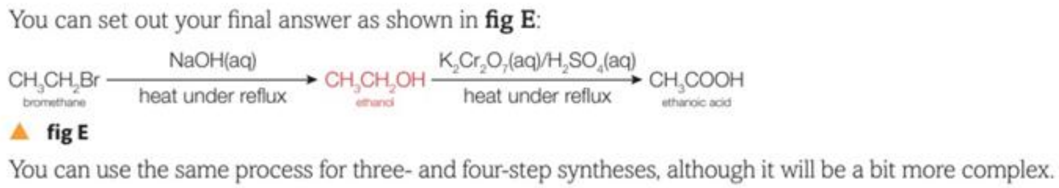
• Now look at the starting compound, bromoethane, to see if it can be converted into one of these compounds. If necessary, write the formulae of the compounds that bromoethane can easily be converted into. These include ethanol, ethene and ethylamine.

• You should now be able to see a clear two-step route from bromoethane to ethanoic acid. In this case, the route is to convert bromoethane into ethanol, and then to convert ethanol into ethanoic acid.

• Finally, you need to add the reagents and conditions required for each step. To convert bromoethane into ethanol you need to add aqueous sodium hydroxide and heat under reflux.

To convert ethanol into ethanoic acid you need to add potassium dichromate(VI) and dilute sulfuric acid and heat under reflux.

You can set out your final answer as shown in fig E:



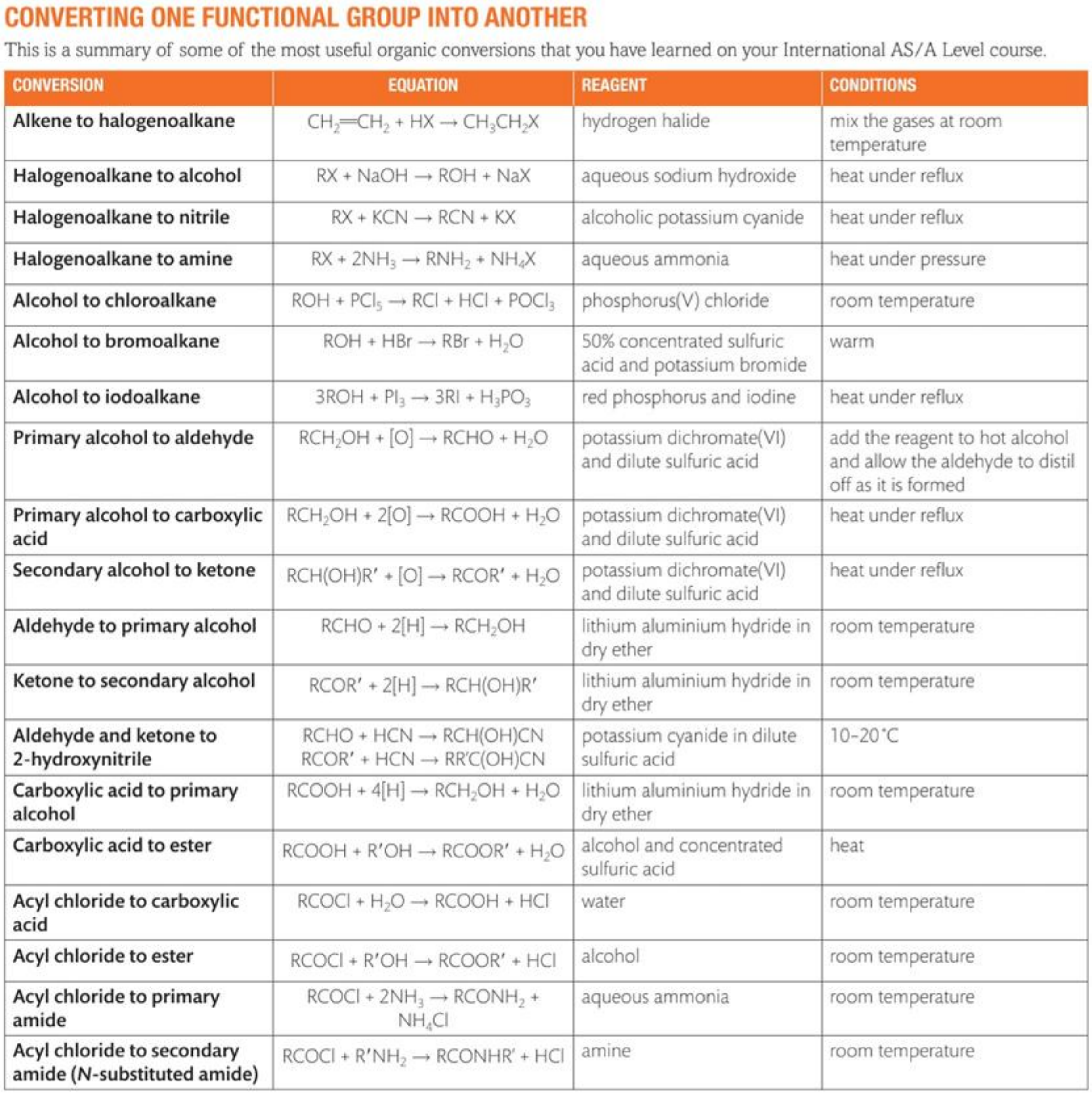
You can set out your final answer as shown in **fig E**:

$$CH_3CH_2Br \xrightarrow[\text{heat under reflux}]{NaOH(aq)} CH_3CH_2OH \xrightarrow[\text{heat under reflux}]{K_2Cr_2O_7(aq)/H_2SO_4(aq)} CH_3COOH$$

**fig E**

You can use the same process for three- and four-step syntheses, although it will be a bit more complex.

# 20A 3 HAZARDS, RISKS AND CONTROL MEASURES



## CONVERTING ONE FUNCTIONAL GROUP INTO ANOTHER

This is a summary of some of the most useful organic conversions that you have learned on your International AS/A Level course.

| CONVERSION | EQUATION | REAGENT | CONDITIONS |
|---|---|---|---|
| **Alkene to halogenoalkane** | $CH_2{=}CH_2 + HX \rightarrow CH_3CH_2X$ | hydrogen halide | mix the gases at room temperature |
| **Halogenoalkane to alcohol** | $RX + NaOH \rightarrow ROH + NaX$ | aqueous sodium hydroxide | heat under reflux |
| **Halogenoalkane to nitrile** | $RX + KCN \rightarrow RCN + KX$ | alcoholic potassium cyanide | heat under reflux |
| **Halogenoalkane to amine** | $RX + 2NH_3 \rightarrow RNH_2 + NH_4X$ | aqueous ammonia | heat under pressure |
| **Alcohol to chloroalkane** | $ROH + PCl_5 \rightarrow RCl + HCl + POCl_3$ | phosphorus(V) chloride | room temperature |
| **Alcohol to bromoalkane** | $ROH + HBr \rightarrow RBr + H_2O$ | 50% concentrated sulfuric acid and potassium bromide | warm |
| **Alcohol to iodoalkane** | $3ROH + PI_3 \rightarrow 3RI + H_3PO_3$ | red phosphorus and iodine | heat under reflux |
| **Primary alcohol to aldehyde** | $RCH_2OH + [O] \rightarrow RCHO + H_2O$ | potassium dichromate(VI) and dilute sulfuric acid | add the reagent to hot alcohol and allow the aldehyde to distil off as it is formed |
| **Primary alcohol to carboxylic acid** | $RCH_2OH + 2[O] \rightarrow RCOOH + H_2O$ | potassium dichromate(VI) and dilute sulfuric acid | heat under reflux |
| **Secondary alcohol to ketone** | $RCH(OH)R' + [O] \rightarrow RCOR' + H_2O$ | potassium dichromate(VI) and dilute sulfuric acid | heat under reflux |
| **Aldehyde to primary alcohol** | $RCHO + 2[H] \rightarrow RCH_2OH$ | lithium aluminium hydride in dry ether | room temperature |
| **Ketone to secondary alcohol** | $RCOR' + 2[H] \rightarrow RCH(OH)R'$ | lithium aluminium hydride in dry ether | room temperature |
| **Aldehyde and ketone to 2-hydroxynitrile** | $RCHO + HCN \rightarrow RCH(OH)CN$<br>$RCOR' + HCN \rightarrow RR'C(OH)CN$ | potassium cyanide in dilute sulfuric acid | 10–20 °C |
| **Carboxylic acid to primary alcohol** | $RCOOH + 4[H] \rightarrow RCH_2OH + H_2O$ | lithium aluminium hydride in dry ether | room temperature |
| **Carboxylic acid to ester** | $RCOOH + R'OH \rightarrow RCOOR' + H_2O$ | alcohol and concentrated sulfuric acid | heat |
| **Acyl chloride to carboxylic acid** | $RCOCl + H_2O \rightarrow RCOOH + HCl$ | water | room temperature |
| **Acyl chloride to ester** | $RCOCl + R'OH \rightarrow RCOOR' + HCl$ | alcohol | room temperature |
| **Acyl chloride to primary amide** | $RCOCl + 2NH_3 \rightarrow RCONH_2 + NH_4Cl$ | aqueous ammonia | room temperature |
| **Acyl chloride to secondary amide (*N*-substituted amide)** | $RCOCl + R'NH_2 \rightarrow RCONHR' + HCl$ | amine | room temperature |

| CONVERSION | EQUATION | REAGENT | CONDITIONS |
|---|---|---|---|
| **Nitrile to primary amine** | $RCN + 4[H] \rightarrow RCH_2NH_2$ | lithium aluminium hydride in dry ether | room temperature |
| **Nitration of benzene** | $C_6H_6 + HNO_3 \rightarrow C_6H_5NO_2 + H_2O$ | concentrated nitric acid and concentrated sulfuric acid | heat under reflux between 50 and 60 °C |
| **Sulfonation of benzene** | $C_6H_6 + H_2SO_4 \rightarrow C_6H_5SO_3H + H_2O$ | fuming sulfuric acid | 40 °C |
| **Bromination of benzene** | $C_6H_6 + Br_2 \rightarrow C_6H_5Br + HBr$ | liquid bromine with iron (to form iron(III) bromide) | dry and room temperature |
| **Friedel-Crafts alkylation of benzene** | $C_6H_6 + RX \rightarrow C_6H_5R + HX$ | halogenoalkane | dry, in the presence of anhydrous aluminium halide |
| **Friedel-Crafts acylation of benzene** | $C_6H_6 + RCOCl \rightarrow C_6H_5COR + HCl$ | acyl chloride | dry, in the presence of anhydrous aluminium chloride |

## SAFETY IN CHEMISTRY LABORATORIES

Incidents that cause harm to people are rare in school and college chemistry laboratories. One of the reasons for this is that all laboratories must consider the hazards of carrying out chemistry experiments and use safe methods of working.

## HAZARDS, RISKS AND CONTROL MEASURES

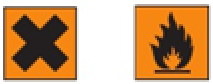
Chemical hazards are determined by the properties of the substance and its usage. Water is generally considered safe, but boiling water can cause harmful effects if it comes into contact with skin. Dilute hydrochloric acid, a corrosive substance, can cause harm if it gets into the eyes, requiring eye protection. Understanding the difference between **hazard and risk** is crucial in managing chemical hazards.

| SYMBOL | MEANING | |
|---|---|---|
|  | Health hazard | includes warnings on skin rashes, eye damage and ingestion |
|  | Corrosive | can cause skin burns and permanent eye damage |
|  | Flammable | can catch fire if heated or comes into contact with a flame |
|  | Acute toxicity | can cause life-threatening effects, even in small quantities |

**table A**

## HAZARD WARNING SYMBOLS

More recently, symbols (sometimes called pictograms) have been used as labels for bottles to identify the actual hazard of the substance inside. The actual symbols have changed over the years, and you may still see older ones used, especially those in squares with an orange background, such as these:



## Control measures

The laboratory manager is responsible for identifying risks and hazards, and prescribing appropriate control measures. Students are responsible for following guidance. Health and safety information from organizations may be found in the laboratory. Students should be familiar with this information when planning experiments or synthesis.

DANGER

OVEN CLEANER

HAZARD STATEMENTS:
Causes severe skin burns and eye damage

PRECAUTIONARY STATEMENTS:
- Wear Protective gloves, protective clothing, eye protection, face protection
- Wash thoroughly after handling
- IF SWALLOWED: Rinse mouth Do NOT induce vomiting
- IF IN EYES: Rinse catiously with water for several minutes. Remove contact lenses if present and easy to do continue rinsing
- IF ON SKIN:Remove/Take off immediately all contaminated clothing. Rinse skin with water/shower
- Dispose of contents/container in accordance with local regulations.

SEE SDS FOR MORE INFORMATION

▲ **fig A** You will also find examples of hazards, risks and control measures in your home.

## APPARATUS

Consider risks associated with apparatus, such as replacing mercury thermometers with spirit or digital thermometers, using electrical heating mantles, using ground glass joints for safer setup, and considering appropriate clamp and stand support.

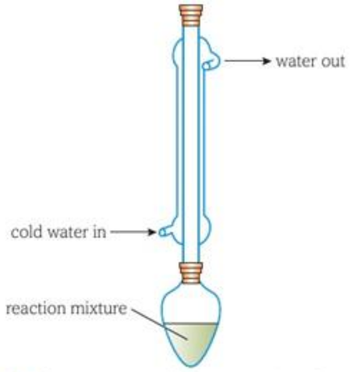
# 20A 4 PRACTICAL TECHNIQUES IN ORGANIC CHEMISTRY: PART 1

## DIFFERENT PRACTICAL TECHNIQUES

In the preparation and purification of any organic compound, there are likely to be several techniques used. Each preparation or synthesis will use one or more of the techniques in this section and the next. Most of them have been covered in Book 1, but all are included in these sections for completeness.

## HEATING UNDER REFLUX

Some reactions involving organic compounds are slow at room temperature, so the obvious thing to do is heat the reaction mixture. However, as many organic compounds are volatile, there is a risk that they will escape from the reaction mixture during the heating process. The normal way to prevent this happening is to heat the reaction mixture in a flask fitted with a reflux condenser (fig A).

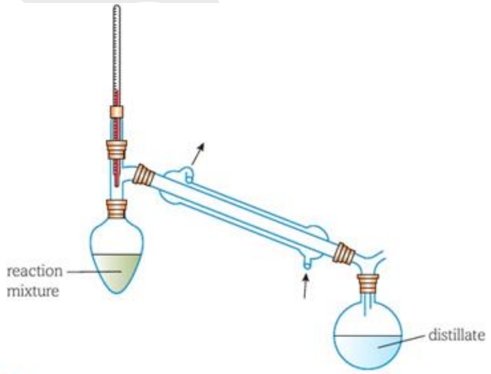


▲ **fig A** Apparatus for heating under reflux.

# METHODS OF SEPARATION

## SIMPLE DISTILLATION

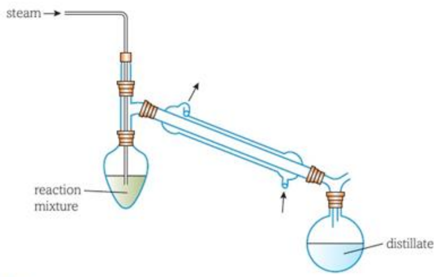
Distillation is a separation technique used to extract a liquid product from a reaction mixture with a lower boiling temperature. It involves heating an impure liquid in a flask connected to a condenser, with the lowest boiling liquid evaporating first. A thermometer monitors the vapor temperature, indicating a different compound is being distilled.



▲ **fig B** Apparatus for simple distillation.

### Steam distillation

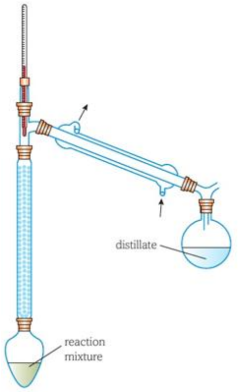
Steam distillation is a method to separate an insoluble liquid from an aqueous solution by passing steam into a reaction mixture. This process forms a separate layer, allowing both the insoluble and aqueous solution to evaporate. The advantage is that the insoluble liquid is removed at a lower temperature, reducing the chance of decomposition, such as phenylamine, which has a boiling temperature of 184°C.



▲ **fig C** Apparatus for steam distillation.

## FRACTIONAL DISTILLATION

Fractional distillation uses the same apparatus as simple distillation, but with a fractionating column between the heating flask and the still head (fig D). The column is usually filled with glass beads or pieces of broken glass. These act as surfaces on which the vapour leaving the column can condense, and then be evaporated again as more hot vapour passes up the column. Effectively, the vapour experiences several repeated distillations as it passes up the column, which provides a better separation.



▲ **fig D** Apparatus for fractional distillation.

# 20A 5 PRACTICAL TECHNIQUES IN ORGANIC CHEMISTRY: PART 2

## MORE METHODS OF SEPARATION

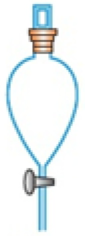
### SOLVENT EXTRACTION

As the name suggests, this method involves using a solvent to remove the desired organic product from the other substances in the reaction mixture. Several solvents can be used, but the choice depends mainly on these features:

- The solvent added should be immiscible (i.e. does not form a mixture) with the solvent containing the desired organic product.
- The desired organic product should be much more soluble in the added solvent than in the reaction mixture.

The method is this:

- Place the reaction mixture in a separating funnel (fig A), and then add the chosen solvent - it should form a separate layer.
- Place the stopper in the neck of the funnel and gently agitate the contents of the funnel for a short while (put a finger on the stopper, invert, open the tap, agitate in a circular motion, close the tap and return the funnel to its normal position).
- Allow the contents to settle into two layers.
- Remove the stopper and open the tap to allow the lower layer to drain into a flask, and then do the same to allow the upper layer to drain into a separate flask.



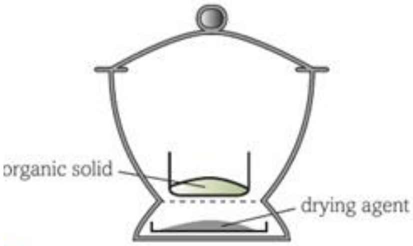
▲ **fig A** Apparatus for separating immiscible liquids.

### WASHING

Washing is a technique used to remove impurities from both solids and liquids. It involves using water, organic solvents, or sodium carbonate solution to dissolve impurities. The mixture is filtered, and the solid is added to the solvent. The liquid is mixed with the chosen solvent, shaken in a separating funnel, and drained into separate containers.

### DRYING

No special technique is needed to dry an organic solid - it just needs to be left in a warm place or in a desiccator with a suitable drying agent (fig B).



▲ **fig B** Drying an organic solid in a desiccator

Organic liquids are prepared using inorganic reagents in aqueous solutions. Water can dissolve in the liquid, requiring a drying agent to remove impurities. Common drying agents include anhydrous metal salts like calcium chloride, magnesium sulfate, and sodium sulfate. These salts form hydrated salts that absorb water as crystallization water. The agent is added to the liquid, agitated, and left to dry. The liquid changes from cloudy to clear when water is removed.

## FILTRATION

It is likely that after preparation, an organic solid will need to be filtered at some stage. This always happens as part of recrystallisation, so the two pieces of apparatus (Buchner and Hirsch funnels) that are normally used will be considered under the next heading. The use of a vacuum pump means that these methods are described as filtration under reduced pressure.

### *RECRYSTALLISATION*

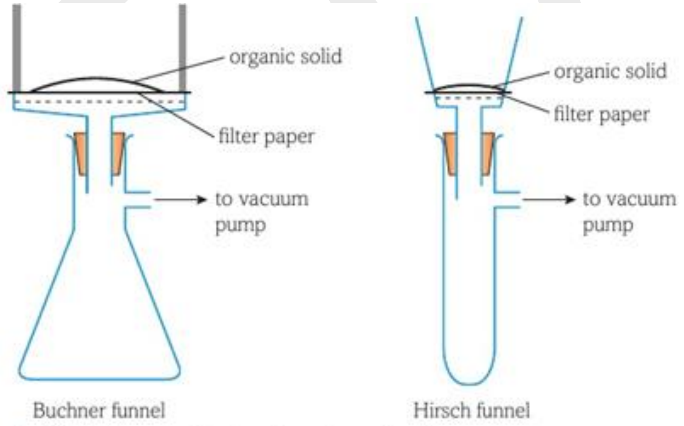
When an organic solid has been prepared, it is likely to need purification. A traditional way of removing impurities is the technique of recrystallisation. The principle behind this technique is that a solid compound is dissolved in a suitable solvent that can dissolve all or most of any impurities but very little of the compound being purified. The steps used in a typical purification are:

- Add the impure solid to a conical flask.
- Add some of the chosen solvent and warm until the mixture nears the boiling temperature of the mixture.
- If there is still some undissolved solid, add further solvent and warm until the mixture boils again.
- Continue adding further solvent and heating until all of the soluble solid has dissolved.
- If insoluble impurities are present, then hot filtration could be done using fluted filter paper in a heated funnel.
- Allow the liquid to cool until crystals of the organic solid have formed.
- More crystals can be obtained by cooling the solution below room temperature in an ice bath.

The mixture is then filtered to remove soluble impurities using a Buchner funnel or a Hirsch funnel (fig C).

- The crystals are washed with a small amount of ice-cold solvent and then dried in a desiccator

or warm oven.



▲ **fig C** Apparatus for filtration under reduced pressure.

## TESTING FOR PURITY

Having prepared an organic compound, a simple test can be performed to give an indication of whether the compound is pure. If the compound is a solid, then its melting temperature can be measured. If it is a liquid, then its boiling temperature can be measured.

## DETERMINATION OF MELTING TEMPERATURE

The melting temperature of a solid can be measured by comparing it with the known value of the pure compound. Traditional methods involve placing the solid in a capillary tube attached to a thermometer bulb, but these are often replaced by electrical devices.

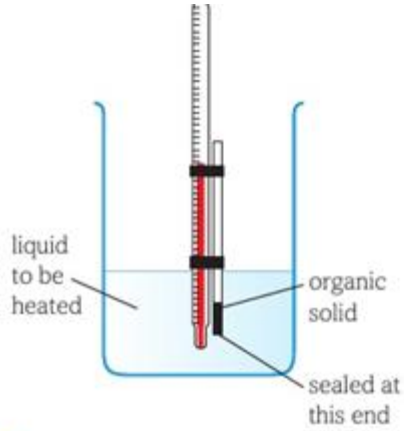


**fig D** Apparatus for determination of melting temperature.

## DETERMINATION OF BOILING TEMPERATURE

Pure organic compounds' boiling temperatures are available in data books and online, and can be estimated by comparing the measured value with the known value. However, accurate measurements may not be enough to confirm purity. Different organic compounds can have the same boiling temperature, such as 1-chloropentane and 2-methylpropan-1-ol boiling at 108°C. The apparatus used depends on the volume of liquid and its flammability.