**1** The following instructions are from an experimental procedure for the preparation of cyclohexene from cyclohexanol and concentrated phosphoric acid.
Read these instructions and answer the questions that follow.

1 Place 25 $cm^3$ of cyclohexanol into a round-bottomed flask with some porous pot to act as anti-bumping granules. Add 10 $cm^3$ of concentrated phosphoric acid carefully while shaking the flask. Cool the flask under the tap if it gets too hot.
Make sure the reagents are thoroughly mixed.

2 Set up an apparatus for simple distillation using this flask.

3 Warm the flask, gently at first, for about 15 minutes. Then increase the heating so that cyclohexene begins to distil over. Collect the fraction that distils below 95 °C.

(a) State the purpose of the anti-bumping granules.

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**(1)**

(b) Name the part of the distillation apparatus where cyclohexene vapour is changed back into a liquid.
Draw a simple diagram of this part of the apparatus.

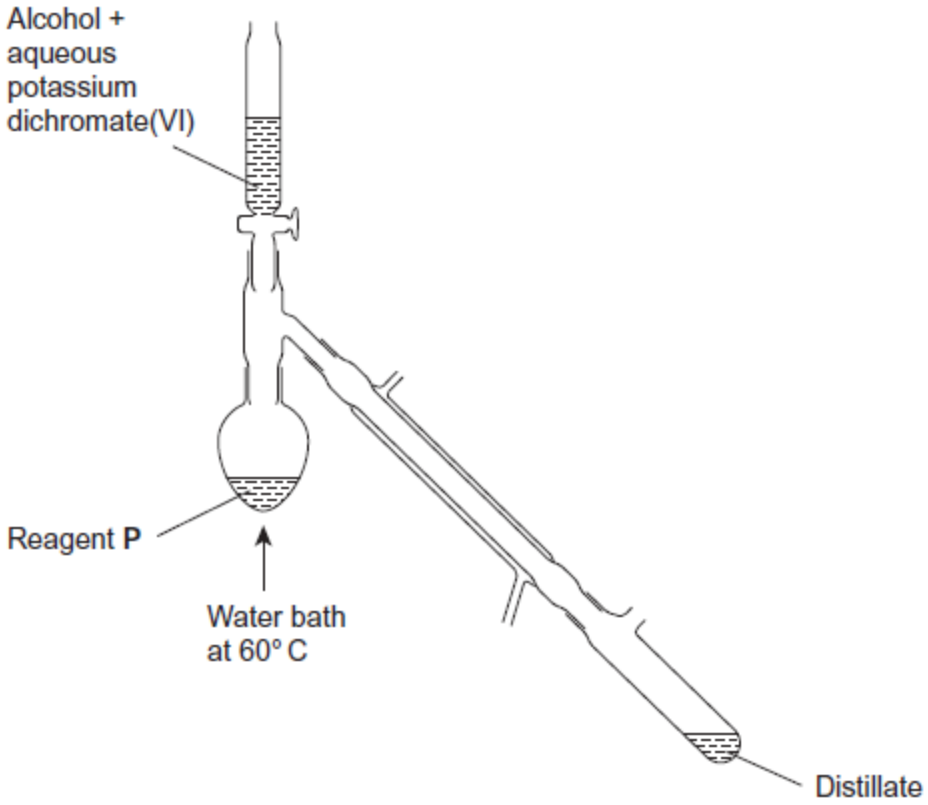
Name .....

Diagram

**(2)**
**(Total 3 marks)**

**2** This question concerns the oxidation of a primary alcohol.

The experiment was carried out using the distillation apparatus shown in the diagram.
The oxidation product was distilled off as soon as it was formed.




(a) Suggest the identity of reagent **P**.

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**(1)**

(b) State the chemical change that causes the solution in the flask to appear green at the end of the reaction.

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**(1)**

(c) Give **one** reason why using a water bath is better than direct heating with a Bunsen burner.

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**(1)**

(d) Suggest a reagent that could be used to confirm the presence of an aldehyde in the distillate.
State the observation you would expect to make if an aldehyde were present.

Reagent .....

Observation .....

**(2)**
**(Total 5 marks)**

**3** Ethanol can be oxidised by acidified potassium dichromate(VI) to ethanoic acid in a two-step process.

ethanol ⟶ ethanal ⟶ ethanoic acid

(a) In order to ensure that the oxidation to ethanoic acid is complete, the reaction is carried out under reflux.

Describe what happens when a reaction mixture is refluxed and why it is necessary, in this case, for complete oxidation to ethanoic acid.

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**(3)**

(b) Write a half-equation for the overall oxidation of ethanol into ethanoic acid.

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**(1)**

(c) The boiling points of the organic compounds in a reaction mixture are shown in the following table.

| **Compound** | ethanol | ethanal | ethanoic acid |
|---|---|---|---|
| **Boiling point / °C** | 78 | 21 | 118 |

Use these data to describe how you would obtain a sample of ethanal from a mixture of these three compounds. Include in your answer a description of the apparatus you would use and how you would minimise the loss of ethanal. Your description of the apparatus can be either a description in words or a labelled sketch.

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**(5)**

(d) Use your knowledge of structure and bonding to explain why it is possible to separate ethanal in this way.

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**(2)**

(e) A student obtained a sample of a liquid using the apparatus in part **(c)**.

Describe how the student could use chemical tests to confirm that the liquid contained ethanal and did **not** contain ethanoic acid.

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**(5)**
**(Total 16 marks)**

**4** (a) A chemist discovered four unlabelled bottles of liquid, each of which contained a different pure organic compound. The compounds were known to be propan-1-ol, propanal, propanoic acid and 1-chloropropane.

Describe four **different** test-tube reactions, one for each compound, that could be used to identify the four organic compounds.
Your answer should include the name of the organic compound, the reagent(s) used and the expected observation for each test.

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*(Extra space)* .....

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**(8)**

(b) A fifth bottle was discovered labelled propan-2-ol. The chemist showed, using infrared spectroscopy, that the propan-2-ol was contaminated with propanone.

The chemist separated the two compounds using column chromatography. The column contained silica gel, a polar stationary phase.

The contaminated propan-2-ol was dissolved in hexane and poured into the column. Pure hexane was added slowly to the top of the column. Samples of the eluent (the solution leaving the bottom of the column) were collected.

- Suggest the chemical process that would cause a sample of propan-2-ol to become contaminated with propanone.
- State how the infrared spectrum showed the presence of propanone.
- Suggest why propanone was present in samples of the eluent collected first (those with shorter retention times), whereas samples containing propan-2-ol were collected later.

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*(Extra space)* .....

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**(4)**
**(Total 12 marks)**

**5** Ethanoic acid, propyl ethanoate and propan-1-ol are all colourless liquids. Esters do **not** give a positive result with any of the usual tests for functional groups.

State how you could use chemical tests to show the presence of ethanoic acid and propan-1-ol in a mixture of the acid, the alcohol and the ester.

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**(Total 4 marks)**

**1** (a) To prevent vigorous boiling / uneven boiling / bubbling vigorously

*Reference to an effect on 'reaction' here loses this mark.*

**1**

(b) Condenser

*Accept 'condensation chamber' or 'condensation tube'.*

**1**

Should show effective water jacket and central tube

*If a flask is also drawn then the condenser must be at an appropriate angle.*

*Apparatus must clearly work.*

*Ignore direction of water flow.*

*Diagram must have a clear flow of vapour and water eg unblocked central tube or flow indicated by arrows.*

**1**

**[3]**

**2** (a) $H_2SO_4$

*Allow $H_3PO_4$ or HCl*

**1**

(b) Dichromate / Cr(VI) reduced or Cr(III) formed.

*Allow $Cr^{6+}$ and $Cr^{3+}$*

**1**

(c) The alcohol is flammable

*Allow enables temperature to be controlled*

**1**

(d) Tollens'

**1**

Silver mirror
**OR** Fehling's
Red precipitate
**OR** Benedict's
Red precipitate

**1**

**[5]**

**3** (a) A mixture of liquids is heated to boiling point for a prolonged time

**1**

Vapour is formed which escapes from the liquid mixture, is changed back into liquid and returned to the liquid mixture

**1**

Any ethanal and ethanol that initially evaporates can then be oxidised

**1**

(b) $CH_3CH_2OH + H_2O \longrightarrow CH_3COOH + 4H^+ + 4e^-$

**1**

(c) Mixture heated in a suitable flask / container

*A labelled sketch illustrating these points scores the marks*

**1**

With still head containing a thermometer

**1**

Water cooled condenser connected to the still head and suitable cooled collecting vessel

**1**

Collect sample at the boiling point of ethanal

**1**

Cooled collection vessel necessary to reduce evaporation of ethanal

**1**

(d) Hydrogen bonding in ethanol and ethanoic acid or no hydrogen bonding in ethanal

**1**

Intermolecular forces / dipole-dipole are weaker than hydrogen bonding

**1**

(e) Reagent to confirm the presence of ethanal:

Add Tollens' reagent / ammoniacal silver nitrate / aqueous silver nitrate followed by 1 drop of aqueous sodium hydroxide, then enough aqueous ammonia to dissolve the precipitate formed

**OR**

Add Fehling's solution

**1**

Warm

*M2 and M3 can only be awarded if M1 is given correctly*

**1**

Result with Tollen's reagent:

Silver mirror / black precipitate

**OR**

Result with Fehling's solution:

Red precipitate / orange-red precipitate

**1**

Reagent to confirm the absence of ethanoic acid

Add sodium hydrogencarbonate or sodium carbonate

**1**

Result; no effervescence observed; hence no acid present

1

*M5 can only be awarded if M4 is given correctly*

**OR**

Reagent; add ethanol and concentrated sulfuric acid and warm

Result; no sweet smell / no oily drops on the surface of the liquid,

hence no acid present

**[16]**

**4** (a) **If 2 stage test for one compound, award no marks for that compound, eg no mark for ROH or RX to alkene then $Br_2$ test. If reagent is wrong or missing, no mark for that test; if wrong but close/incomplete, lose reagent mark but can award for correct observation. In each test, penalise each example of wrong chemistry, eg $AgClr_2$**

propan-1-ol

acidified
potassium
dichromate

sodium

Named acid + conc $H_2SO_4$

named acyl chloride

$PCl_5$

M1

1

(orange) turns green

effervescence

Sweet smell

Sweet smell /misty fumes

Misty fumes

M2

1

propanal

add Tollens or Fehlings / Benedicts

acidified
potassium
dichromate

Bradys or 2,4-dnph

*if dichromate used for alcohol cannot be used for aldehyde*

M3

**1**

Tollens: silver mirror or Fehlings/ Benedicts: red ppt

(orange) turns green

Yellow or orange ppt

M4

**1**

propanoic acid

Named carbonate/ hydrogencarbonate

water and UI (paper)

Named alcohol + conc $H_2SO_4$

sodium or magnesium

$PCl_5$

*if sodium used for alcohol cannot be used for acid*

M5

**1**

effervescence

orange/red

Sweet smell

effervescence

Misty fumes

*if $PCl_5$ used for alcohol cannot be used for acid*

M6

**1**

1-chloro propane

NaOH then acidified $AgNO_3$

$AgNO_3$

*If acidification missed after NaOH,*
*no mark here but allow mark for observation*

M7

**1**

white ppt

white ppt

M8

**1**

(b) oxidation (of alcohol by oxygen in air)

M1

**1**

absorption at <u>1680 -1750</u> (due to C=O)

*Must refer to the spectrum*

M2

**1**

comparison of polarity of molecules or correct imf statement:
propanone is less polar OR propan-2-ol is more polar
OR propanone has dipole-dipole forces
OR propan-2-ol has hydrogen bonding

M3

**1**

about attraction to stationary phase or solubility in moving phase
Propan-2-ol has greater affinity for stationary phase or vice versa
OR propanone is more soluble in solvent/moving phase or vice versa

M4

**1**

**[12]**

**5** Identification of acid by suitable method eg named indicator, named carbonate, specified reactive metal

*Ignore any reference to the smell of the ester.*

**1**

with expected results

*Do not allow the use of any instrumental method eg i.r. or n.m.r.;*
*must be a <u>chemical</u> test.*

**1**

Identification of alcohol by suitable method eg oxidation by acidified potassium dichromate(VI)

**1**

with expected results

**1**

**[4]**