Course 2- Practical Organic Chemistry

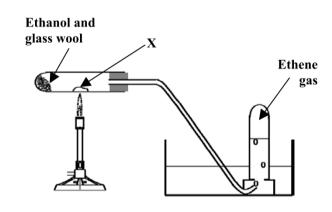
Question 1

- **2.** Ethene gas can be prepared from ethanol in a school laboratory.
 - (a) Draw a labelled diagram showing the arrangement of apparatus and the reagents used in the preparation and collection of the ethene. (11)
 - (b) It is important to be aware of the possibility of a 'suck-back' occurring when carrying out this procedure.
 - (i) At what stage in the procedure is a 'suck-back' most likely to occur?
 - (ii) Give one possible consequence of a 'suck-back' occurring.
 - (iii) How could a 'suck-back' be avoided?
 - (c) Describe how you could test the gas produced for unsaturation. (9)
 - (d) Write a balanced equation for the preparation of ethene from ethanol. (6)
 - (e) When ethanol is converted to ethene by this method, a 60% yield can be expected.

Assuming this percentage yield, what is the maximum number of 75 cm³ test tubes of ethene gas that could be collected at room temperature and pressure when 2.4 cm³ of ethanol, density 0.8 g cm⁻³, react? (15)

Question 2

- 2. The diagram shows the experimental set-up used by a group of students to prepare a sample of ethene from ethanol and to collect the ethene produced.
 - (a) What is the function of the glass wool? (5) Identify the solid **X** and describe its appearance. (6)
 - (b) State and explain **two** safety precautions which should be observed when carrying out the student experiment.



(9)

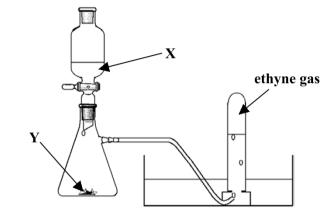
(6)

- (c) Write a balanced equation for the reaction involved in this preparation.
- (d) If the ethene produced is bubbled through an acidified solution of potassium manganate(VII), the solution is decolorised showing that ethene is *unsaturated*. What is meant by the term *unsaturated*? Describe how you would carry out another test to confirm that ethene is unsaturated. (12)

(12)

(e) Describe the flame that would be observed when a combustion test is carried out on a sample of ethene gas. Write a balanced equation for the combustion of ethene in excess oxygen. (9)

- 2. The diagram shows an apparatus that can be used for the preparation of ethyne gas, C_2H_2 . A liquid X is dropped onto the solid Y and the gas collected in test tubes as shown.
 - (a) Identify the liquid \mathbf{X} and the solid \mathbf{Y} . (8)
 - (b) Describe the appearance of the solid \mathbf{Y} . (3)
 - (c) Write a balanced equation for the reaction between **X** and **Y** producing ethyne. (6)
 - (d) What is observed when a sample of ethyne gas is burned in air?
 Write a balanced equation for the combustion of ethyne in oxygen.
 - (e) Ethyne, C₂H₂, is described as an *unsaturated* hydrocarbon.



Describe a test you could carry out to show that ethyne is *unsaturated*.

Write an equation for the reaction taking place. Name the organic product. (18)

(f) The common name for ethyne gas, C_2H_2 , is acetylene. Give **one** major use of the gas. (6)

- 2. Ethene can be prepared in the school laboratory using the arrangement of apparatus shown in Diagram 1. Ethyne can be prepared in the school laboratory using the arrangement of apparatus shown in Diagram 2.
- (a) Give the name or chemical formula of the solid X used in the preparation of ethene.What is the colour of this solid? (5)
- (b) Write a balanced equation for the reaction involved in the preparation of ethene. What term describes this type of reaction? (6)
- (c) State **three** precautions that should be observed when carrying out the preparation of ethene by this method. (9)
- (d) Give the name or formula of the solid Y used in the preparation of ethyne in the school laboratory.
 Describe the appearance of this solid.
- (e) Both ethene and ethyne are described as *unsaturated*.

 What does this mean? Describe a test you could carry out on a sample of either gas to show that it is unsaturated.

 What would you observe during the test? (9)
- Both ethene and ethyne can be burned in air.
 What is the most noticeable difference seen when these combustions are carried out in a school laboratory?
 Write a balanced equation for the complete combustion of either gas.
- (g) Give (i) a major use of ethene,
 - (ii) a major use of ethyne. (6)

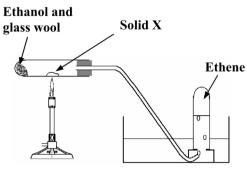
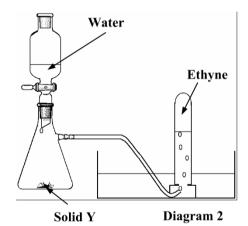
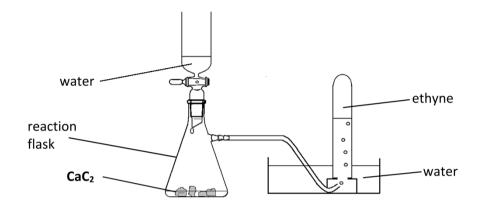


Diagram 1



- **2.** A student prepared ethene and ethyne gases and compared their properties.
 - (a) The apparatus shown was used to prepare and collect ethyne.
 - (i) What was observed in the reaction flask as the water was dropped onto the calcium carbide (CaC_2)?
 - (ii) Explain why the first few test-tubes of gas collected were discarded. (6)



- (b) (i) Draw a labelled diagram of a suitable arrangement of apparatus and chemicals for the preparation and collection of ethene.
 - (ii) State and explain two safety precautions that should be taken when carrying out the experiment. (24)
- (c) Combustion tests were carried out on samples of ethene and ethyne.
 - (i) Compare the observations made in the two tests.
 - (ii) Write a balanced equation for the complete combustion of ethyne in oxygen. (9)
- (d) Name a reagent used to test the gases for unsaturation. (3)
- (e) The mass of calcium carbide consumed in the preparation of ethyne, in an apparatus like that shown above, was 3.2 g. The equation for the reaction is as follows:

$$CaC_2 + 2H_2O \rightarrow Ca(OH)_2 + C_2H_2$$

Calculate the maximum theoretical number of ethyne molecules produced in this reaction. (8)

2. (a) Draw a labelled diagram of a suitable arrangement of apparatus and of chemicals for the preparation and collection over water of samples of pure ethene gas.

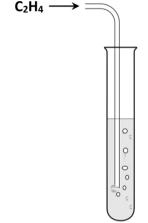
When carrying out this preparation, state one way of avoiding

(i) a suck-back of cold water into the reaction vessel,

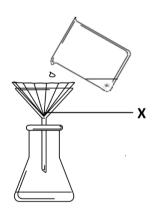
(b) Some ethene was bubbled through a reagent as shown in the diagram on the right.

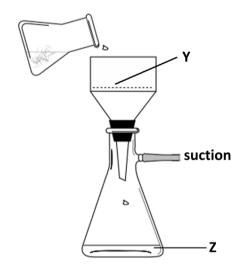
What colour change is observed when the reagent used is

- (i) dilute acidified KMnO₄,
- (ii) bromine water?
- (iii) Name the organic reaction type that occurs in the case of the bromine reagent.
- (iv) Identify an organic product of the reaction between ethene and the bromine solution. (12)



(c) A student practiced the technique of recrystallisation using benzoic acid containing small quantities of two solid impurities: water-soluble potassium chloride (KCI) and black, insoluble charcoal (C). The two filtration stages in the recrystallisation of the benzoic acid using water as the solvent are shown in the diagrams below in the order in which they are carried out.





(i) Identify the substance(s) collected at locations **X**, **Y** and **Z**.

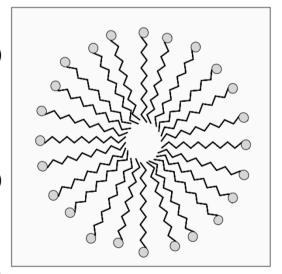
What steps are carried out to maximise the yield of pure benzoic acid,

- (ii) between the first and second filtrations,
- (iii) after the second filtration?
- (iv) How would you expect the melting point value of a recrystallised sample of benzoic acid to differ from that of an impure sample? (21)

2. Sodium cocoate soap is manufactured by the base hydrolysis of tri-ester molecules in coconut oil. The following is a balanced equation for the hydrolysis of a tri-ester molecule in coconut oil.

To make sodium cocoate soap, a student mixed some coconut oil, an excess of solid **NaOH**, water and ethanol as solvents, and anti-bumping material in a round-bottomed flask and refluxed the mixture gently for about 20 minutes. The student then removed the ethanol by distillation and transferred the residue of the reaction mixture into a beaker containing brine. The student separated the soap that precipitated from the solution in the beaker and washed it with a small volume of ice-cold water. The product was tested by shaking a small sample of it with deionised water in a stoppered test-tube to confirm that a lather formed.

- (a) (i) Why was the reaction mixture refluxed?
 - (ii) Suggest a suitable heating method for refluxing the reaction mixture and then distilling off the ethanol.
 - (iii) Justify the suitability of your suggested heating method.
 - (iv) Why is it desirable to remove all the ethanol? (14)
- (b) (i) Name the co-product of the reaction.
 - (ii) What is brine?
 - (iii) Explain the function of the brine in the procedure. (12)
- (c) (i) Describe how the solid soap was separated from the solution in the beaker.
 - (ii) Where is the excess **NaOH** at this stage of the procedure? (9)
- (d) The diagram represents the arrangement of a number of soap molecules in water.
 Explain why one end of a soap molecule is described as water loving (hydrophilic).



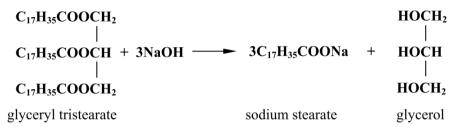
(e) When 0.03 moles of the tri-ester shown in the equation above are hydrolysed, what is the theoretical yield, in grams, of CH₃(CH₂)₁₀COONa?

2. A student prepared a sample of soap in the school laboratory. The experiment was carried out in the four stages illustrated on the previous page. At Stage 1, using a water bath, the student refluxed for approximately 20 minutes 4.45 g of glyceryl tristearate (an animal fat) together with an excess of sodium hydroxide pellets, anti-bumping material and about 30 cm³ of ethanol. The reaction shown in the following balanced equation took place.

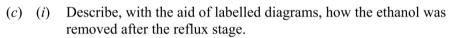
The apparatus was then allowed to cool and rearranged for Stage 2, distillation, again using a water bath. After distillation, the contents of the distillation flask were decanted or washed into a beaker containing brine – Stage 3. Filtration was used in Stage 4 to isolate the soap which was then thoroughly washed.

- (a) What is the purpose of refluxing in Stage 1 of the preparation?Name the type of reaction that occurred during this stage.(8)
- (b) What substance was removed by distillation in Stage 2? (3)
- (c) Explain the function of the brine in Stage 3. (6)
- (d) Why was it necessary to wash the soap thoroughly in Stage 4?How should the student have washed the soap? (6)
- (e) Draw the structure or give the name of the co-product of the reaction.Where was the co-product located at the end of the process? (9)
- (f) Given that the sodium hydroxide was in excess, calculate the maximum yield in grams of soap that could have been obtained in this preparation. (12)
- (g) Suggest, with reference to its structure, how a soap like sodium stearate can dissolve both the non-polar oils *and* the ionic salts in sweat from the skin. (6)

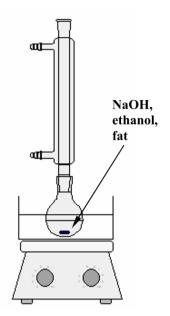
2. To prepare soap, a student refluxed 8.9 g of glyceryl tristearate (obtained from animal fat), 2 g of sodium hydroxide pellets and 30 cm³ of ethanol, together with a few anti-bumping granules, using the apparatus shown on the right. At the end of the experiment 7.0 g of pure, dry soap were isolated. The balanced equation for the reaction is as follows:



- (a) Write the systematic (IUPAC) name for glycerol. (5)
- (b) Why was the reaction mixture heated under reflux?Name the type of reaction that occurred during the reflux.What was the purpose of the ethanol? (12)

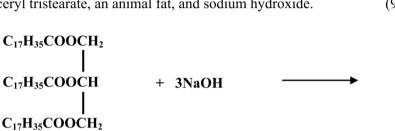


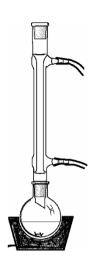
- (ii) How was the soap isolated from the other substances left in the reaction mixture?
- (iii) After isolating the soap, how was it purified and dried?
- (d) Given that the sodium hydroxide was in excess, calculate the percentage yield of soap (sodium stearate). (12)



(21)

- 2. A sample of soap was prepared in the laboratory by refluxing a mixture of approximately 5 g of animal fat, 2 g of sodium hydroxide pellets (an excess) and 25 cm³ of ethanol in an apparatus like that drawn on the right.
 - (a) Why was the reaction mixture refluxed? Name the *type* of reaction which occurs during the reflux stage of the preparation. (8)
 - (b) Complete and balance the equation below for the reaction between glyceryl tristearate, an animal fat, and sodium hydroxide. (9)





(12)

(6)

glyceryl tristearate

- (c) What is the purpose of the ethanol? Why is it desirable to remove the ethanol after reflux? Describe with the aid of a labelled diagram how you would remove the ethanol after the reflux stage of the experiment.
- (d) Describe how a pure sample of soap was obtained from the reaction mixture. (9)
- (e) At the end of the experiment, what is the location
 - (i) of the second product of the reaction,
 - (ii) of the excess sodium hydroxide?
- (f) What would you observe, upon shaking, if a little of the soap prepared in this experiment is added to (i) a test tube containing deionised water,
 (ii) a test tube containing mineral water from a limestone region?

- 2. Soap is produced by the hydrolysis of vegetable and animal fats.
 - (a) What is the principal chemical difference between vegetable and animal fats?

(5)

A sample of soap was prepared in a school laboratory as follows: Approximately 3 g of lard (animal fat), 2 g of sodium hydroxide pellets (an excess), and 25 cm³ of ethanol were placed in a round-bottomed flask. A condenser was fitted to the flask and the mixture was refluxed gently for 20 minutes (Diagram 1).

Following the reflux, the apparatus was allowed to cool slightly and the arrangement of the apparatus was changed so that the ethanol could be removed by distillation (Diagram 2).

Diagram 1

The residue from the distillation flask was then dissolved in a minimum of hot water and the solution decanted into 75 cm³ of brine. The soap was then isolated.

- (b) Apart from the lard, sodium hydroxide and ethanol, what else should be added to the reaction flask prior to the reflux? Why was the mixture refluxed? Why was the ethanol added?
- (c) Why was it desirable to remove the ethanol after the reflux?
- (d) Why was a <u>minimum</u> of hot water used to dissolve the residue from the distillation? What is brine?
- (e) Describe how the soap could be isolated from the mixture of soap and brine. Give <u>one</u> precaution that helps to ensure that the soap is free of sodium hydroxide.

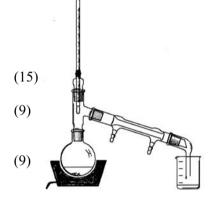


Diagram 2

(12)

2. One molecule of phenylmethanol (benzyl alcohol) can be oxidised to one molecule of benzoic acid by potassium manganate(VII) in basic conditions as shown in the reaction scheme below.

- (a) Describe the appearance of phenylmethanol at room temperature. (5)
- (b) State the colour change when phenylmethanol was heated gently with potassium manganate(VII) solution to which sodium carbonate had been added.

Identify **A** and **B** in the reaction scheme that were added to allow separation of the benzoic acid from the other substances after the oxidation was complete.

What changes were observed in the reaction vessel on addition of these two chemicals and as cooling occurred?

State the changes in the oxidation number of manganese during the experiment. (24)

(c) After isolation by filtration, the benzoic acid crystals were purified by recrystallisation.

State one way of maximising the yield of the recrystallisation process. The melting point range of the benzoic acid crystals before recrystallisation was 112 - 118 °C.

How would recrystallisation have affected the melting point range? (9)

(d) A student oxidised 2.7 cm³ of phenylmethanol (density 1.04 g cm⁻³) and obtained 1.83 g of benzoic acid after recrystallisation. Assuming that phenylmethanol was the limiting reactant, calculate the percentage yield of benzoic acid. (12)

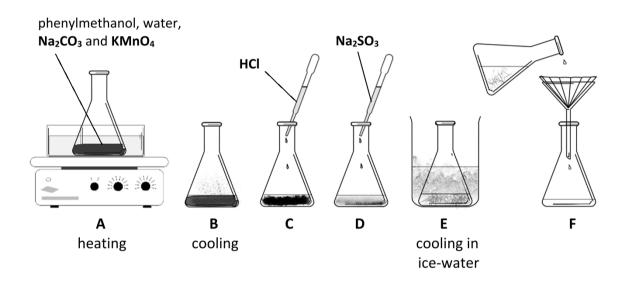
2. Benzoic acid is synthesised by the oxidation of phenylmethanol (benzyl alcohol) with KMnO₄ in alkaline conditions. The overall balanced equation for the oxidation reaction is:

$$3C_6H_5CH_2OH + 4KMnO_4 \rightarrow 3C_6H_5COOH + 4MnO_2 + H_2O + 4KOH$$

The stages of the synthesis are illustrated in diagrams A to F below.

Stages A and B are the oxidation stages.

Concentrated **HCl** is added at stage **C** and concentrated **Na₂SO₃** solution is added at stage **D**. Benzoic acid crystals are collected at stage **F**.



- (a) (i) Describe the appearance of phenylmethanol at room temperature.
 - (ii) Name a suitable piece of apparatus to measure accurately 1.5 cm³ of phenylmethanol.

(5)

- (b) How does the appearance of the reaction mixture change when the phenylmethanol is heated along with potassium manganate(VII) solution to which sodium carbonate has been added? (6)
- (c) Describe what is observed during stages **D** and **E**.

 Explain these observations. (12)
- (d) (i) Name a suitable technique to purify the benzoic acid crystals collected at stage **F**.
 - (ii) Describe how you measured the melting point of a sample of benzoic acid crystals. (12)
- (e) A student used a solution containing 3.16 g of potassium manganate(VII) to oxidise 1.5 cm³ phenylmethanol (density 1.04 g cm⁻³) as described above.
 - (i) Show by calculation whether there is sufficient potassium manganate(VII) to oxidise all the phenylmethanol according to the balanced equation above.
 - (ii) Find the theoretical yield in grams of benzoic acid. (15)

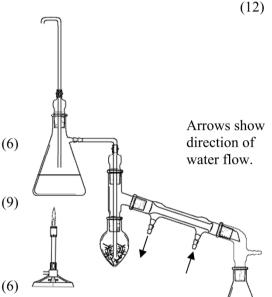
- 2. In a practical examination, chemistry students were required to perform a number of tasks in a laboratory. They had access to all the necessary reagents and glassware and also to the required safety equipment and clothing.
 - (a) How could a student have carried out a simple chemical test to confirm that a colourless liquid sample was ethanoic acid and not ethanol? (5)
 - (b) A sample of ethene gas was supplied in a stoppered test tube. Describe fully how the gas could have been shown to be unsaturated. (12)
 - (c) Describe with the aid of a labelled diagram how a student could have used chromatography to separate a mixture of indicators.
 - (d) One of the tasks in the practical examination was to measure the melting points of two benzoic acid samples (**A** and **B**) and to use the results to determine which was the purer sample. The melting points obtained by one of the students were as follows: sample $\mathbf{A} = 117 120 \,^{\circ}\text{C}$; sample $\mathbf{B} = 120 121 \,^{\circ}\text{C}$.

Which was the purer sample? Justify your answer.

The students were required to recrystallize the impure benzoic acid. What solvent should they have used for the recrystallization? Explain why this solvent is suitable. (9)

(e) The diagram shows a steam distillation apparatus assembled **incorrectly** by one of the students.

Identify the flaw in the assembly and state how it should have been rectified.



2. (a) Reflux is a widely used technique in organic chemistry.

Identify an experiment from your course where you refluxed a mixture.

Draw a <u>fully labelled</u> diagram of the reflux apparatus used in this experiment.

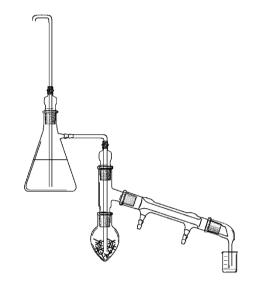
What happened to the liquid in the flask during reflux?

How did refluxing this mixture help bring the reaction to completion?

(30)

(9)

- (b) The diagram shows an apparatus suitable for steam distillation.
 - (i) What natural product did you extract in the school laboratory using steam distillation?
 - (ii) What was the appearance of the material collected during the steam distillation?
 - (iii) What substance distilled across along with the natural product?
 - (iv) Identify **one** safety feature of the apparatus drawn. Explain how this feature contributes to the safe use of the apparatus. (20)



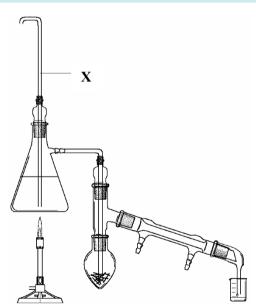
Question 16

Steam distillation, using an apparatus like that shown, is a technique used to isolate an organic substance from plant material. The principle of this technique is that the boiling point of a mixture of two *immiscible liquids* is below the boiling points of both pure liquids. This allows the organic substance to be isolated at temperatures below 100 °C and avoids the delicate organic molecules being damaged at high temperatures.

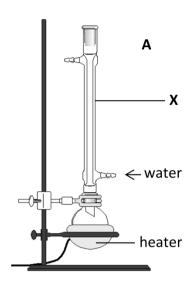
- (c) What is meant by the term *immiscible liquids*? (3)
- (d) Name a substance you isolated by steam distillation in the school laboratory and the plant material from which it was extracted. (6)
- (e) Explain the function of the tube labelled X. (6)
- (f) Describe the appearance of the distillate collected.

 Name or describe briefly a technique that could be used to separate the organic substance from the water.
- (g) In a steam distillation experiment 20.0 g of plant material were heated in the presence of steam.

 Only 0.250 g of pure organic liquid was obtained. Calculate the percentage yield. (5)



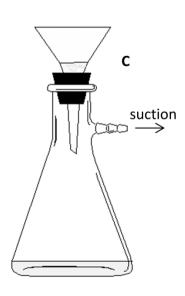
- **2.** (a) Diagram **A** shows an arrangement used to reflux the reaction mixture in an experiment to prepare a sample of soap.
 - (i) Name the piece of glassware labelled X.
 - (ii) What is the purpose of refluxing a reaction mixture?
 - (iii) Identify the two reactants and the solvent initially present in the flask.
 - (b) Diagram B shows a separating funnel in use in a liquid-liquid extraction of clove oil from the emulsion produced in a steam distillation of cloves.
 - (i) What is an emulsion?
 - (ii) Name a suitable organic solvent for use in the liquid-liquid extraction of clove oil.
 - (iii) Other than wearing eye-protection, gloves and a laboratory coat, and tying back long hair, state one safety precaution that should be taken when using a separating funnel.
 - (iv) How is the clove oil isolated following the liquid-liquid extraction?
 - (v) Steam distillation and liquid-liquid extraction were used to isolate 0.15 cm³ of clove oil (density 1.05 g cm⁻³) from 5.0 g of cloves.
 What was the percentage yield, by mass, of clove oil? (18)
 - (c) A sample of benzoic acid, containing a small quantity of sodium chloride as the only impurity, was purified by recrystallisation. The impure crystals were dissolved in the minimum amount of boiling water. This solution was cooled thoroughly and the crystals that formed were separated by suction filtration as shown in diagram C. The crystals were then dried.
 - (i) Why was there no need, in this recrystallisation, to carry out a filtration of the hot solution?
 - (ii) State one advantage of suction filtration over gravity filtration.
 - (iii) Explain how you could verify that the recrystallised benzoic acid was purer than the original sample.



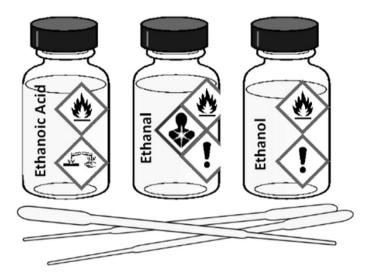
(18)

(14)





2. Ethanoic acid, ethanal and ethanol are three colourless organic substances, all liquids at $18\,^{\circ}$ C. A student was provided with small, pure samples of each of these substances in closed sample bottles.



- (a) The student placed a short strip of freshly-sanded magnesium ribbon into a test tube containing a few drops of ethanoic acid in approximately 10 cm³ of water and swirled the test tube.
 - (i) What was observed?
 - (ii) Write formulae for both products of the reaction that occurred. (11)
- (b) Describe a chemical test to show that ethanal can be very easily oxidised. (9)
- (c) The student was asked to prepare a small quantity of ethene from ethanol using the reaction shown by the balanced equation below. A suitable catalyst and a heat source were provided.

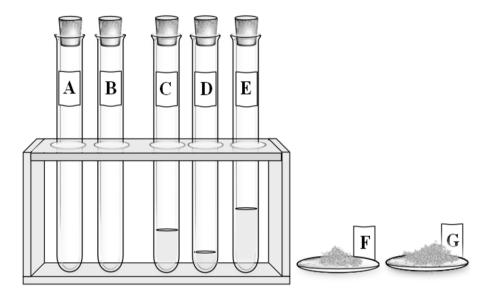
$$C_2H_5OH \rightarrow C_2H_4 + H_2O$$

- (i) Draw a diagram of an arrangement of apparatus for the preparation and collection of ethene. Indicate clearly on your diagram the location of the ethanol, the catalyst, the heat source, and the ethene produced.
- (ii) Identify the catalyst and describe its appearance at the beginning of the preparation.
- (iii) Explain clearly why a risk of hot glassware shattering, due to rapid cooling, is associated with this preparation.How can this risk be minimised?
- (*iv*) A student prepared ethene, starting with 2.9 cm³ of ethanol (density 0.8 g cm⁻³). Calculate the volume of ethene gas produced in 26% yield when measured at room temperature and pressure. (30)

2. The two test-tubes on the left in the diagram contain pure samples of **A** and **B**, different gaseous hydrocarbons, one of which is unsaturated.

The pure liquids C, D and E in the other test-tubes are samples of ethanal, ethanoic acid and eugenol, but not necessarily in that order.

The crystalline solids **F** and **G** are two different benzoic acid samples.



- (a) Describe how to chemically test sample \mathbf{A} or \mathbf{B} for unsaturation. (9)
- (b) (i) At room temperature (20 °C) it was observed that **D** evaporated more quickly than either **C** or **E**.
 Based on this observation deduce the identity of liquid **D**.
 Describe a chemical test to confirm that **D** can be very easily oxidised. (12)
 - (ii) Some water was added to liquids **C** and **E** and the test-tubes were stoppered and shaken vigorously. After the test-tubes were allowed to stand, a white emulsion was observed in the test-tube containing **C** and a colourless solution in the test-tube containing **E**. Identify **C** and **E**.

A small volume of cyclohexane was then added to the test-tube containing **C**. The test-tube was stoppered and again shaken vigorously and the stopper loosened. What was observed after the test-tube was again allowed to stand? (9)

- (iii) A piece of freshly-sanded magnesium ribbon was then added to the test-tube containing **E**. Write a balanced equation for the reaction that took place. (6)
- (c) (i) Describe with the aid of a labelled diagram a method used to measure the melting point of a sample of benzoic acid.
 - (ii) The melting-point range of **F** is lower and broader than that of **G**. Which is the purer benzoic acid sample?
 - (iii) Name a laboratory technique that could be used to purify impure benzoic acid.

(14)