(b) The preparation of an organic liquid compound (e.g. the preparation of an ester). It is also expected that candidates will be aware of the procedures involved in the purification and re-distillation at an appropriate temperature of the product obtained.

The Preparation of Ethyl Ethanoate

Reaction

1) Mix 50cm^3 of ethanol and 50cm^3 of *glacial* ethanoic acid thoroughly in a 250cm^3 round-bottomed flask.

2) Add *slowly with cooling* and shaking 10cm^3 of *concentrated sulphuric acid*.

3) Ensure that the mixture is *homogeneous*, then fit the flask with a *reflux water-condenser* and boil the mixture gently *for 10 minutes*.

Separation of the product

4) Rearrange the position of the condenser for distillation, and distil off about two-thirds of the mixture.

5) Transfer the distillate to a separating funnel and add about 25 cm^3 of 30% *sodium carbonate solution*. Stopper the funnel, invert it, and shake, *opening the tap from time to time*.

6) Allow the two layers to separate and carefully run off and reject the lower layer, ensuring that the sodium carbonate is removed as completely as possible.

Purification of the Product (you do not need to carry out this part)

7) Add some saturated calcium chloride solution to the ethyl ethanoate, shake well, and remove the lower aqueous layer.

8) Pour the ethyl ethanoate into a beaker, add a few lumps of solid anhydrous calcium chloride and shake well. Decant the ethyl ethanoate into a flask.

9) Then decant the liquid into a *clean, dry* 100 cm^3 round-bottom flask, add some *anti-bumping granules*, and arrange for distillation including a 0-100^{o}C thermometer in the apparatus. The distilling flask should be placed in a cold water bath, which is gradually heated. *The ether that is always formed* in this reaction will distil off at 35-40^{o}C, and may be discarded. Continue to heat, and collect the fraction that boils between 74^o and 79^{o}C.

Questions:

- **Why must the concentrated sulphuric acid be added slowly and with cooling?**
- **Why is concentrated sulphuric acid used?**
- **Why must the mixture be homogeneous?**
- **What is a reflux water-condenser?**
- **Why is the reaction comparatively slow?**
- **What is the function of the sodium carbonate solution?**
- **Why do you need to open the tap from time to time?**
- **What is the function of the calcium chloride solution?**
- **What is the function of the solid calcium chloride?**
- **Why should the distillation flask be clean and dry?**
- **What is the nature and purpose of anti-bumping granules?**
- **Why is an ether formed in this reaction?**

- **Explain the meaning of the term *glacial* as applied to ethanoic acid.**
- Glacial ethanoic acid is pure ethanoic acid CH_3COOH. It is called glacial because on cooling it forms glassy crystals.

- **Why must the concentrated sulphuric acid be added slowly and with cooling?**
- The concentrated sulphuric acid on dilution gives out a lot of heat; the slow addition with cooling is necessary to avoid splashing if the mixture gets hot.
- **Why is concentrated sulphuric acid used?**
- Concentrated sulphuric acid is a ***catalyst*** for the esterification reaction

$$CH_3CH_2OH + CH_3COOH \rightleftharpoons CH_3COOCH_2CH_3 + H_2O$$

- **Why must the mixture be homogeneous?**
- Concentrated sulphuric acid is much denser than any of the other reagents. If not well mixed initially, the solution is liable to get too hot and boil uncontrollably when mixing occurs later in the reaction.
- **What is a reflux water-condenser?**
- A reflux water condenser is a Liebig condenser arranged vertically above the reaction flask; vapors are condensed and returned to the flask, the contents of which can therefore be boiled for long periods without any loss of material.
- **Why is the reaction comparatively slow?**
- Most organic reactions are slow since they involve the breaking of strong covalent bonds. The proportion of molecular collisions that have the necessary activation energy is usually fairly low.

- **What is the function of the sodium carbonate solution?**
- The distillate contains traces of ethanoic acid and perhaps some sulphuric acid. Sodium carbonate solution removes this.
- **Why do you need to open the tap from time to time?**
- The neutralisation with sodium carbonate produces carbon dioxide gas; opening the tap releases this and avoids a build-up of pressure that might blow the stopper out of the funnel.

- **What is the function of the calcium chloride at this stage of the preparation?**
- The crude ethyl ethanoate contains traces of ethanol; the calcium chloride solution removes this since it complexes with the ethanol

- **What is the function of the calcium chloride at this stage of the preparation?**
- The solid calcium chloride is a dehydrating agent; it removes any remaining traces of water from the product.

- **Why should the distillation flask be clean and dry?**
- You have just dried the mixture; you don't want it wet again.
- **What is the nature and purpose of anti-bumping granules?**
- Anti-bumping granules are small pieces of silica; broken unglazed pottery works as well. This provides a nucleus on which gas bubbles grow, therefore avoiding the sudden production of large gas bubbles which can lead to 'bumping'. This, properly called succussion, causes liquid to splash over into the condenser and therefore produce an impure product. Severe bumping can even lead to loss of material through vents or can blow

a distillation apparatus apart.

- **Why is an ether formed in this reaction?**
- An ether is formed because sulphuric acid can cause the elimination of a molecule of water between two alcohol molecules. The water causes the ionisation of the sulphuric acid:

$CH_3CH_2OH + HOCH_2CH_3 + H_2SO_4 \rightarrow$

$CH_3CH_2OCH_2CH_3 + H_3O^+ + HSO_4^-$