**Apparatus:**

Conc. sulphuric acid 15cm3

Ethanol 30cm3

Glacial ethanoic acid

Saturated sodium carbonate solution

Anhydrous calcium chloride lumps

Calcium chloride hydrate (.6H2O)

Anti-bumping granules

0 – 360oC thermometers

Gloves

Boiling tubes

IR disc / IR spectrometer

**Method for making ethyl ethanoate:**

1. Pour 15cm3 of ethanol into a 100cm3 round bottomed flask and 15cm3 ethanoic acid. Put this into a water bath; carefully add 10cm3 concentrated sulphuric acid and swirl. Wipe off the water from the flask with tissue paper. Add 3 anti bumping granules.
2. Fit a two-necked adapter to the round bottomed flask. Place a separating funnel into the adapter so that liquid can run vertically down the flask.
3. Set up the apparatus for distillation with a 0-100oC thermometer and a small conical flask to receive the ester. Use a heating mantle to heat the round bottom flask.
4. Heat the mixture using the heating mantle. INCREASE THE TEMPERATURE SLOWLY otherwise the mixture will bubble over – this is dangerous.
5. The ester will distil over at between 65o and 75oC. Keep distilling until no more ester comes over.

**Method for purification:**

1. Remove the distillate and wash with 20cm3 saturated aqueous sodium carbonate solution in a separating funnel. Discard the lower aqueous layer.
2. Shake the impure ester with a solution made by dissolving 10g hydrated calcium chloride in 10cm3 water. Separate and discard the lower layer.
3. Stand the ester over a few lumps of anhydrous calcium chloride until the liquid clears – 20 minutes. The liquid may then be filtered or decanted, and distilled in clean dry *Quickfit*.

**Testing the boiling point:**

Pure ethyl ethanoate boils at 73oC.

This is the same temperature at which you should have seen the product was distilled.

What temperature did your product come off? This is your boiling point.

**Infrared spectrum:**

Use a pipette to take a drop of your sample. Place it in between two IR discs, and place into the IR machine.

A spectrum will be produced. Label all peaks fully, in particular pay attention to the C=O.

How do you know your sample does **not** contain any of the reactants? (hint: think about OH peak, COOH peak, are these present in your product? Should they be?)