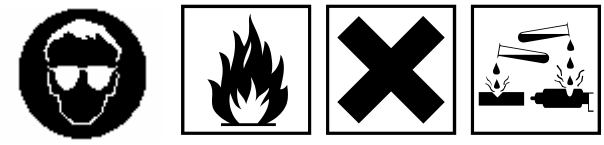
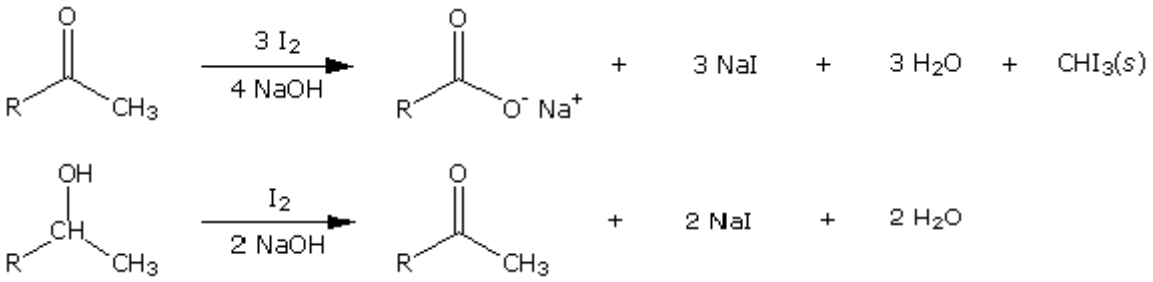
Preparation of triiodomethane (iodoform)

Aims



Secondary alcohols with an adjacent methyl groups can be oxidised to methyl ketones, as shown below.



You will prepare the solid product CHI_3 (triiodomethane or "iodoform") from propanone.

Apparatus

Goggles	25cm^3 measuring cylinder	2 x boiling tube	Filter paper
Bench mat	50cm^3 measuring cylinder	Test tube rack	Spatula
10cm^3 measuring cylinder	250cm^3 beaker	Buchner funnel and flask	Teat pipette

Reagents

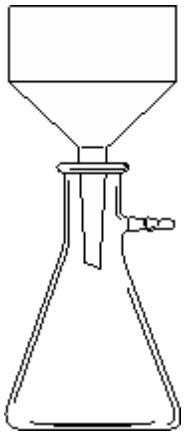
Propanone	10% potassium iodide solution	10% sodium chlorate(I) solution
Ethanol	2M sodium hydroxide solution	

Methods

Part One Reaction to produce triiodomethane

1. Use pipettes and measuring cylinders to put the following into a 250cm^3 conical flask:
   - 1cm^3 propanone
   - 40cm^3 10% potassium iodide solution
   - 16cm^3 2M sodium hydroxide solution
   - 40cm^3 10% sodium chlorate(I) solution
2. Mix the contents of the flask well by swirling it over 3 to 4 minutes.

Part Two Filtration



3. Set up a Buchner funnel and flask as shown in the diagram. Connect the flask to the water pump and put a piece of filter paper into the Buchner funnel. Lightly dampen the filter paper with de-ionised water (this helps it to stick to the funnel). Turn on the water pump.
4. Carefully pour the reaction mixture from the flask into the Buchner funnel. When the liquid has been drawn through the funnel, carefully add de-ionised water to wash the crystals. Allow the water to drain thoroughly.

Part Three Recrystallisation

5. Use a spatula to scrape your washed solid from the filter paper into a boiling tube.

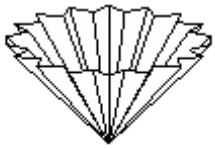
6. Boil a beaker of water. If you have used a Bunsen burner to do this, turn the flame off. Put a boiling tube of ethanol into the boiling water (it may begin to boil; this is safer than using a Bunsen burner to heat the ethanol directly because ethanol is flammable). Put the boiling tube containing your solid product into the water bath.

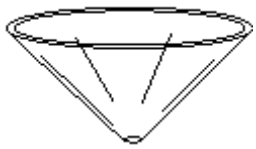
7. Add a few drops of **hot** ethanol to your product. If the solid has not dissolved in the hot ethanol, add a few drops more. Repeat this until the solid just dissolves. This ensures that only the minimum amount of solvent has been used.

   If you have scraped off some filter paper with your crystals, this will not dissolve, of course.
   If you have bits of insoluble impurities like this in your solution, you will have to filter the mixture using the method described below. If your solution is clear, **do not** filter it.

   To filter off insoluble impurities:

   Line a stemless funnel with fluted filter paper, and pour hot water through it to preheat it. Pour your solution into the filter funnel. Crystals may form in the stem of the funnel if it cools too much – wash these through with a minimal volume of hot ethanol.





8. Allow the solution to cool. As it cools, solid triiodomethane will crystallise out, and any **soluble** impurities should remain in solution.

9. Use the Buchner funnel and flask again to filter off the crystals. Wash the crystals on the filter paper using **cold** ethanol. Remove the filter paper with the crystals on it, and allow it to air dry.

10. Determine the purity of your product by finding its melting point.

Determining a melting point

Aims

To determine the melting point of your triiodomethane (triiodomethane melts between 119°C and 121°C).

Apparatus

Goggles	Melting point apparatus	Bunsen burner
Bench mat	Thermometer	Capillary tube

Methods

1. Carefully seal one end of the capillary tube using a blue flame.
   Add your crystals to the capillary tube – patience!

2. Insert the capillary tube and thermometer into the melting point apparatus and turn it on.

3. Keep watching the crystals until they melt. Note their melting temperature.
   If you have time, repeat your melting point determination but warm slowly near to the melting point.