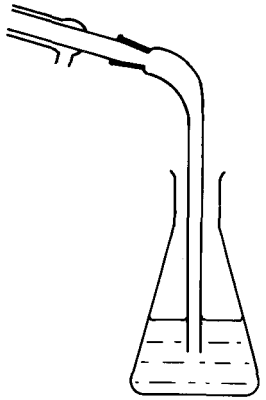
Chapter 12 Small-scale preparations

12.1 1-bromoalkane

Apparatus and reagents

Apparatus for addition reflux (11.4b, 11.4c)
Apparatus for distillation (11.1) modified for collection under water as shown
Measuring cylinder, 25 cm^3 or less
Ethanol, 5 cm^3 **or**
propan-1-ol, 6.5 cm^3 **or**
butan-1-ol, 7.5 cm^3
Potassium bromide, solid, 12 g
Sulphuric acid, concentrated, 10 cm^3
Sodium carbonate, 2 M
Magnesium sulphate, anhydrous



Procedure

In the pear-shaped flask place 12 g potassium bromide, 10 cm^3 water, and the alkanol. Set up the apparatus as in 11.4b or 11.4c, with the flask in a cold water bath. Add 10 cm^3 concentrated sulphuric acid from the tap funnel, shaking the reaction mixture to ensure good mixing. Remove the tap funnel and water bath and reflux the mixture for at least 30 minutes.
After refluxing, allow the apparatus to cool and then rearrange the apparatus for distillation, diagram 11.1, modified as shown. This is to collect volatile product immiscible with water.
Heat the flask gently until the oily drops of distillate cease to come over.
Dismantle the apparatus, clean it, and put it in an oven to dry.
Transfer the distillate to a separating funnel (the tap funnel of the first part of the experiment), discard the upper (aqueous) layer. Add 5 cm^3 sodium carbonate solution and shake. Discard the upper layer. Wash the distillate with 5 cm^3 of water and discard the aqueous layer.
Run the organic layer into a small conical flask or test-tube (150×25 mm). Add small quantities of anhydrous magnesium sulphate and swirl after each addition. When the liquid is clear, stopper the flask or test-tube, and allow to stand for fifteen minutes.
Decant the liquid into the pear-shaped flask and set up the apparatus for distillation, diagram 11.1, collecting the distillate in a specimen bottle.

Product	Boiling range/°C
1-bromoethane	35-40
1-bromopropane	69-74
1-bromobutane	100-103

12.2 Triiodomethane

Apparatus and reagents

Apparatus for recrystallizing (11.6)
Apparatus for melting point determination (11.7)
Suction filtration apparatus
Beaker, 100 cm^3
Ethanol, 1 cm^3
Iodine, 1 g
Potassium iodide, 2 g
Sodium hydroxide, 5 M

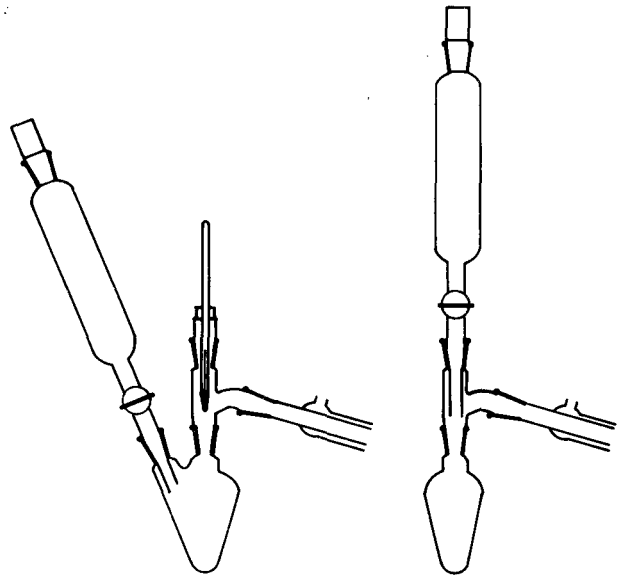
Procedure

Dissolve 2 g potassium iodide in 10 cm^3 of water, add 1 g iodine and dissolve the iodine. Add 1 cm^3 ethanol, then add sodium hydroxide solution until the colour of the iodine disappears. Filter off the yellow precipitate by suction filtration and recrystallize from ethanol, section 11.6.
Check the melting point of the product, 119°C, as in section 11.7.

12.3 Aldehydes

Apparatus and reagents

Apparatus for distillation, (11.1), modified for addition as shown
Beakers, 100 cm^3
Measuring cylinders, 10 cm^3
Propan-1-ol, 5 cm^3 **or**
butan-1-ol, 6 cm^3
Sodium dichromate(VI), 5 g
Sulphuric acid, concentrated, 2 cm^3
Schiffs reagent (13.9)
2,4-DNP reagent (13.4)



Procedure

Add 2 cm^3 concentrated sulphuric acid to 10 cm^3 of water and then add 5 g sodium dichromate(VI). Stir until a solution is formed. Put the alkanol with an equal volume of water in the flask and the dichromate solution in the tap funnel. Set up the apparatus for distillation with addition.
Heat the alkanol-water mixture to boiling and remove the heat source. Add the dichromate solution slowly; a vigorous exothermic reaction occurs. The aldehyde distils over, but it may be necessary to heat the flask occasionally to maintain the transfer of vapour. After the