dichromate solution has been added, heat the flask until the thermometer shows 80-90°C or no more oily drops form in the condenser. Re-assemble the apparatus for distillation (11.1) and return the distillate to the flask. Collect the new distillate in a specimen bottle or measuring cylinder.
Test one drop of the distillate with Schiffs reagent and prepare a derivative of the product by adding 1 cm^3 of 2,4-DNP reagent to 1 cm^3 of the distillate. Filter and dry the product and check its melting point.

Product	Boiling range/°C	Derivative mp/°C
Propanal	47-52	155
Butanal	73-78	126

12.4 Ketones

Apparatus and reagents

Apparatus for distillation with addition as in 12.3
Beakers, 100 cm^3
Measuring cylinders, 10 cm^3
Ice
Propan-2-ol, 5 cm^3 **or**
butan-2-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)
Iodine solution (13.6)
Sodium hydroxide, 2 M

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, collecting the distillate in a flask cooled by an ice-water bath.
Heat the alkanol-water mixture to boiling and remove the heat source. Add the dichromate solution slowly; a vigorous exothermic reaction occurs. The ketone distils over, but it may be necessary to heat the flask occasionally to maintain the transfer of vapour. After the dichromate solution has been added, heat the flask until the thermometer shows 70°C or no more oily drops form in the condenser.
Re-assemble the apparatus for distillation (11.1) and return the distillate to the flask. Collect the new distillate in a dry receiver cooled by an ice-water bath.
Test one drop of the distillate with Schiffs reagent and prepare the 2,4-DNP derivative. Carry out the iodoform reaction on any remaining product.

Product	Boiling range/°C	Derivative mp/°C
Propanone	55 - 60	126
Butanone	79 - 84	116

12.5 Esters

Apparatus and reagents

Apparatus for reflux (11.4a) and distillation (11.1)
Beaker, 100 cm^3
Ethanol, 10 cm^3 **or**
propan-1-ol, 12.5 cm^3
Ethanoic acid, glacial, 5 cm^3
Sulphuric acid, concentrated, 1 cm^3
Calcium chloride, 5 g
Sodium carbonate, 2 M
Magnesium sulphate, anhydrous

Procedure

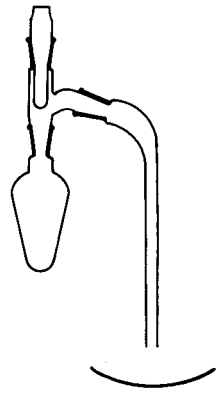
Place the ethanoic acid and ethanol (an excess) in the pear-shaped flask and slowly add 1cm^3 concentrated sulphuric acid. Set up the apparatus for reflux, diagram 11.4a, and reflux in a hot water bath for 30 minutes.
Allow the reaction mixture to cool and then rearrange the apparatus for distillation. The liquid boiling to 85°C (ethyl ethanoate) or 110°C (propyl ethanoate) is collected. Transfer the distillate to a separating funnel and wash with 5 cm^3 sodium carbonate (care — esters are appreciably soluble in water). Release the pressure at intervals. Dissolve 5 g hydrated calcium chloride in 5 cm^3 of water. Add this to the upper ester layer after separation from the aqueous layer and shake to remove excess alkanol. Allow the ester to stand over anhydrous magnesium sulphate in a stoppered test-tube or small flask for about an hour. Thorough drying is necessary, otherwise a constant boiling mixture of low boiling point will distil over.
Meanwhile wash out the distillation apparatus and place in an oven to dry.
Decant the dry solution into the dry flask and set up the apparatus for distillation.

Ester	Boiling range/°C
Ethyl ethanoate	74-79
Propyl ethanoate	100-104

12.6 Ethanamide

Apparatus and reagents

Apparatus for reflux (11.4a)
Apparatus for distillation (11.1)
Apparatus for distillation as shown
Apparatus for recrystallization (11.6)
Apparatus for melting point (11.7)
Watch glasses
Ammonium carbonate, 3 g
Ethanoic acid, glacial, 6 cm^3
Propanone



Procedure

Place 3 g ammonium carbonate in the flask, add 6 cm^3 glacial ethanoic acid and allow the reaction to subside. Reflux gently for 30 minutes. Allow the apparatus to cool and rearrange the apparatus for distillation (11.1) heating the flask directly with a micro-burner. When the thermometer shows 120°C, stop heating and discard the distillate. Rearrange the apparatus as shown in the diagram and collect the distillate on a watch glass.
Resume heating and when the thermometer shows 160°C, place a fresh watch glass under the receiver. The distillate will now consist mostly of ethanamide and should crystallize on the watch glass. In warm conditions it may be necessary to cool in a refrigerator for this to happen. If the product does not solidify, it should be redistilled.
Recrystallize from propanone (11.6) and check the melting point (11.7).

	Boiling range/°C	Melting point/°C
Ethanamide	210-230	82