



A2 4.5/B

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IDENTIFYING AN ESTER



Aim

You are supplied with an unknown ester. You are going to identify the ester by:

- finding its boiling point and
- finding the melting point of the carboxylic acid produced on hydrolysis of the ester.

Safety



Ethanol is flammable – keep the stock bottle away from naked flames



the ester is harmful



Sodium hydroxide solution is very corrosive and is very dangerous in the eyes

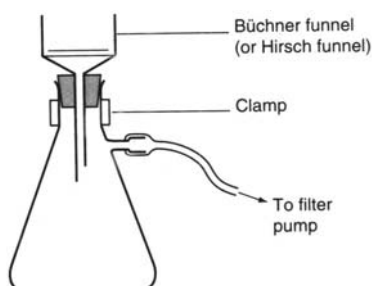
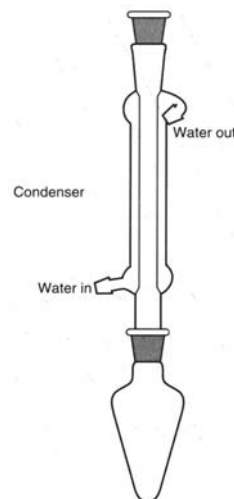
Method

Finding the boiling point of the ester

- 1) Mount a 0-360°C thermometer in the cork provided and adjust its position so that the bulb is about 5 cm from the bottom when placed in the boiling tube.
- 2) Clamp the boiling tube to the retort stand and add the sample of the ester provided and a few anti-bumping granules.
- 3) Heat the liquid gently, using a micro-burner, until it boils. Continue boiling gently and note the maximum reading of the thermometer. This is the boiling point of the ester. (NB – note that the boiling point is above 150°C and any lower reading, due to small amounts of low-boiling impurities, should be ignored).
- 4) Keep the sample for use in the hydrolysis experiment.

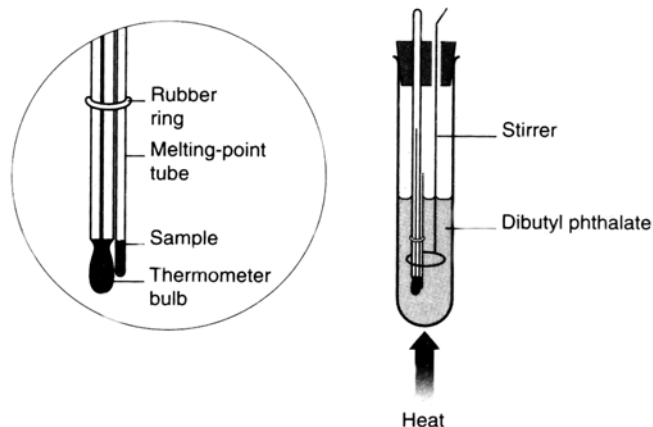
Hydrolysis of the ester

- 5) Transfer 2 cm³ of the ester used above into a quick fit flask.
- 6) Add about 10 cm³ of 2 M sodium hydroxide, about 10 cm³ of ethanol and a few anti-bumping granules.
- 7) Clamp the flask above a Bunsen and insert a reflux condenser.
- 8) Heat the flask gently **without allowing it to boil** for about 5 minutes.
- 9) Heat further to reflux the mixture for a further 15 minutes.
- 10) Allow the contents to cool, remove the reflux condenser, and then decant the liquid from the anti-bumping granules into a beaker.
- 11) Add a few drops of methyl orange and acidify with 2 M hydrochloric acid.
- 12) Filter off the resulting acid under reduced pressure. You should clamp the Buchner flask.



Purification of the resultant acid

- 13) Re-crystallise the acid from the minimum volume of a boiling water.
- 14) Find the mass of your acid.
- 15) Find the melting point of the acid which is above 100°C. To do this place some of the sample in a capillary tube and strap it onto a thermometer. Place the thermometer in a boiling tube half full with dibutyl phthalate and heat, stirring with the rod. Once you know roughly where it melts, repeat the experiment heating to within 30°C of the melting point and then heating very slowly. Record the melting point range.



Data

name of ester	formula	boiling point of ester (°C)	melting point of acid (°C)
ethyl furoate	$C_4H_3OCOOC_2H_5$	197	134
methyl benzenecarboxylate	$C_6H_5COOCH_3$	198	121
methyl 2-methylbenzenecarboxylate	$CH_3C_6H_4COOCH_3$	208	105
ethyl benzenecarboxylate	$C_6H_5COOC_2H_5$	213	121
methyl 3-methylbenzenecarboxylate	$CH_3C_6H_4COOCH_3$	215	112
methyl 4-methylbenzenecarboxylate	$CH_3C_6H_4COOCH_3$	217	181
methyl 2-hydroxybenzenecarboxylate	$HOC_6H_4COOCH_3$	223	155
ethyl 2-methylbenzenecarboxylate	$CH_3C_6H_4COOC_2H_5$	227	105
ethyl 4-methylbenzenecarboxylate	$CH_3C_6H_4COOC_2H_5$	228	181
propyl benzenecarboxylate	$C_6H_5COOC_3H_7$	229	121
ethyl 3-methylbenzenecarboxylate	$CH_3C_6H_4COOC_2H_5$	231	112
ethyl 2-hydroxybenzenecarboxylate	$HOC_6H_4COOC_2H_5$	231	155
methyl 3-chlorobenzenecarboxylate	$ClC_6H_4COOCH_3$	231	158
methyl 2-chlorobenzenecarboxylate	$ClC_6H_4COOCH_3$	234	141

Results

- 1) State the boiling point of your ester.
- 2) State the melting point of the acid.
- 3) State the mass of acid formed.

Questions 1) Use the data table to identify the ester. Give the name and structural formula of the ester.

Name of ester =

Structural formula

2) Write an equation for the hydrolysis of the ester with sodium hydroxide.

3) Write an equation for the reaction with hydrochloric acid at the end of the hydrolysis.

4) Calculate the percentage yield of the acid.

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5) The acid was re-crystallised from water.

Explain why a minimum volume of hot solvent was used.

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Explain why a hot filtration may be necessary.

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