

13 A method of synthesising aspirin is given below using the following steps:

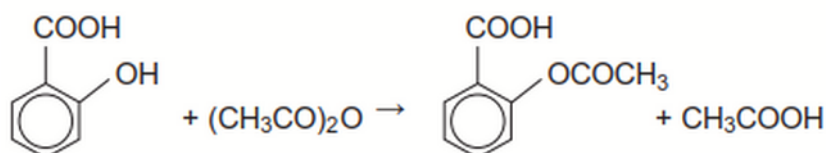
1. Add 7.5 cm³ (an excess) of ethanoic anhydride to 3.0 g of salicylic acid in a conical flask
2. Add eight drops of concentrated phosphoric acid
3. Heat, with stirring, for 20 minutes in a water bath
4. Add 3 cm³ of deionised water to the flask
5. Add 30 cm³ of deionised water and cool to room temperature, allowing the aspirin to crystallise
6. Filter the crystals by Buchner filtration; continue to suck air through the Buchner funnel for five minutes after completion of the filtration

(a) (i) Suggest **two** reasons why ethanoic anhydride is used in this reaction in preference to ethanoic acid.

ethanoic acid forms an equilibrium/incomplete reaction [1]
ethanoic anhydride is more reactive than ethanoic acid [1]

[2]

(ii) Write the equation for the reaction in step 1.



[2]



(iii) Why is concentrated phosphoric acid added?

_____ phosphoric acid acts as a catalyst _____ [1]

(iv) Suggest why water is added in step 4.

_____ The water hydrolyses (any remaining) ethanoic anhydride _____
_____ [1]

(v) Explain why air is sucked through the apparatus for five minutes.

_____ To dry the product _____
_____ [1]

[Turn over



(vi) Describe, giving full experimental detail, the TLC method which can be used to determine whether the reaction is complete after step 3. The solvent is ethyl ethanoate.

In this question you will be assessed on using your written communication skills including the use of specialist scientific terms.

Indicative content

- draw pencil line on TLC plate [1] spot reaction mixture [1]
- on the pencil line spot salicylic acid solution
- place in a beaker with ethyl ethanoate
- (remove plate and) use UV light or a locating reagent
- check if there is a spot for salicylic acid in reaction mixture

[6]

(b) Calculate the percentage yield if 2.3 g of aspirin is obtained (answer to one decimal place).

[3]



(c) Salicylic acid is a bifunctional molecule with a carboxylic acid group and a hydroxyl group attached to the benzene ring. The hydroxyl group displays acidic behaviour.

(i) Suggest why the hydroxyl group attached to a benzene ring is more acidic than the hydroxyl group in aliphatic alcohols.

_____ [2]

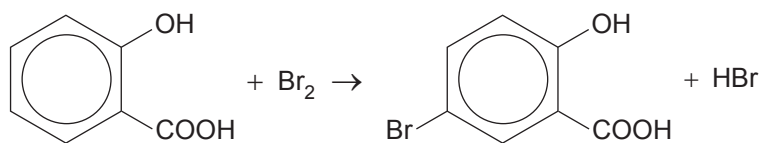
(ii) Write an equation for the reaction of salicylic acid with excess aqueous sodium hydroxide.

_____ [2]

(iii) State why it is preferable to use the sodium salt of aspirin.

_____ [1]

(d) Bromine will give an electrophilic substitution reaction with salicylic acid as shown below:



Salicylic acid will react with bromine without a catalyst being present. This differs from benzene, which requires a metal halide catalyst.

Name a catalyst which is used in the bromination of benzene.

_____ [1]

[Turn over

