# Practical 22 Answers

a) 

b) ethanoic anhydride is in excess; moles of salicylic acid = (5/148)

 so % yield = (mass of aspirin/180)/ (5/148) x 100 =

c) the lower the melting point and the wider the range, the less pure the sample

d) gloves – reactants are corrosive; water bath; avoids overheating; anti-bumping granules; slows down boiling

Recrystallisation – Purifying a Solid Organic Product

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| Method | Reason |
| The solid is dissolved in the minimum possible quantity of hot solvent | The solid should only just dissolve; the solution should be saturated so it crystallises when it cools |
| The solid should be soluble in hot solvent but not in cold solvent | So soluble impurities can be removed when cold and insoluble impurities removed when hot |
| Filter through pre-heated funnel | So that the solution stays hot and does not recrystallize |
| Allow to cool in an ice bath | So that the solid crystallises |
| Filter under reduced pressure | To separate the solid from the solvent |
| Wash the solid with a little cold solvent | To wash off any other soluble impurities |
| Dry the solid | To remove any remaining solvent |

Melting Point Determination – Testing the Purity of the Product

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| --- | --- |
| Method | Reason |
| Pack 1 cm of the solid as densely as possible into the sealed end of the capillary tube | You need enough solid to see it melt clearly, but have to make sure it all fits inside the heater |
| Place the capillary tube in the melting point apparatus. | So you can see the sample through the magnifying glass |
| Ensure that the apparatus heats up very slowly as the expected melting point of the substance approaches | So you can observe the exact temperature at which it starts to melt |
| Record the temperature when the liquid starts to melt | For the purity check, to compare against the known melting point |
| Record the temperature when the liquid finishes melting | For the purity check, to compare against the known melting point |
| Compare the melting point range to the known melting point of the pure solid | To check purity; the higher the melting point and the narrower the range, the more pure the sample |

**Practical 23 Answers**

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| * **Why must the concentrated sulphuric acid be added slowly and with cooling?**

The concentrated sulphuric acid on dilution gives out a lot of heat; the slow addition with cooling is necessary to avoid splashing if the mixture gets hot.* **Why is concentrated sulphuric acid used?**

Concentrated sulphuric acid is a ***catalyst*** for the esterification reaction   CH3CH2OH + CH3COOH http://www.rod.beavon.clara.net/eqm_arrow.gif CH3COOCH2CH3 + H2O |
| * **Why must the mixture be homogeneous?**

Concentrated sulphuric acid is much denser than any of the other reagents. If not well mixed initially, the solution is liable to get too hot and boil uncontrollably when mixing occurs later in the reaction. * **What is a reflux water-condenser?**

A reflux water condenser is a Liebig condenser arranged vertically above the reaction flask; vapors are condensed and returned to the flask, the contents of which can therefore be boiled for long periods without any loss of material. * **Why is the reaction comparatively slow?**

Most organic reactions are slow since they involve the breaking of strong covalent bonds. The proportion of molecular collisions that have the necessary activation energy is usually fairly low. |
| * **What is the function of the sodium carbonate solution?**
* The distillate contains traces of ethanoic acid and perhaps some sulphuric acid. Sodium carbonate solution removes this.
* **Why do you need to open the tap from time to time?**
* The neutralisation with sodium carbonate produces carbon dioxide gas; opening the tap releases this and avoids a build-up of pressure that might blow the stopper out of the funnel.
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| * **Why should the distillation flask be clean and dry?**

You have just dried the mixture; you don't want it wet again. * **What is the nature and purpose of anti-bumping granules?**

Anti-bumping granules are small pieces of silica; broken unglazed pottery works as well. This provides a nucleus on which gas bubbles grow, therefore avoiding the sudden production of large gas bubbles which can lead to 'bumping'. This, properly called succussion, causes liquid to splash over into the condenser and therefore produce an impure product. Severe bumping can even lead to loss of material through vents or can blow a distillation apparatus apart. |