

# Industrial manufacture and testing of an organic solid



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## Introduction -Manufacture of an organic solid in industry

The main process in an industry is, ethanoic acid, toluene and 2-hydroxybenzoic acid combined and then put through a reactor, water is then added, cooling water is added, this is then filtered, the solids are washed, this is then dried, which is then filtered, packaged and aspirin is produced.

(Pearson 2019)

The manufacturing of aspirin is done via the use of a reactor, acetic acid, salicylic acid and the use of an acetic anhydride to complete the reaction. The optimum temperature at which this reaction is based at is 85 degrees Celsius, this temperature is kept constant throughout the reaction by the use of a constant heat source, this allows the reaction to fully complete and not partially complete. After the reaction has fully completed the solution is allowed to cool and therefore can complete the reaction fully and allow the crystals to form. These crystals are then filtered out through the use of a Buchner funnel. These filtered crystals are then washed using distilled water and left to dry. The final product can easily be compressed into tablet form. (Pdf Semantic scholar org 2019).

An organic solid is a chemical compound that is solid at room temperature. In industries there are many ways that organic solids can be manufactured. These include, spray and freeze drying; filter press.

Spray drying is carried out to produce a dry powder, this is done using hot gas. This process involves spraying liquid or slurry into the hot gas, via a

spray nozzle or atomiser and can be done whilst the air is blown in. The hot drying gas can flow with the liquid or against the liquid, however flowing with the liquid results in faster drying time, which can be more effective. This technique is often used in many applications such as medicines, food industry purposes, bone and tooth amalgams, soaps and detergents, textiles ect. (Eurotherm by Schneider Electric, 2019), (Pearson 2019).

Freeze drying incorporates has four stages and is a low temperature dehydration process. It incorporates freezing the product, lowering the pressure and then removing the ice through sublimation. Sublimation is the transition of a substance from a solid to a gas, without going through the liquid phase first. The first stage is pre-treatment, which happens prior to freezing. This may include concentrating the product and or either adding preservatives to it. The second stage is freezing and is greatly significant out of the freeze-drying process. This process is carried out in a freeze-drying machine. It is greatly important to cool the material below its triple point to ensure sublimation will occur. To form larger crystals, the product should be frozen slowly. If not, it can result in a process called annealing. The third stage is called primary-drying. During this phase, the pressure is decreased and for the ice to sublime, in which enough heat is supplied. An estimate of 95% of H<sub>2</sub>O in the material is sublimated. To ensure the material's structure isn't altered, this process is done slowly and can be over several days within the industry. Sublimation Using a partial-vacuum, pressure is controlled, which speeds up sublimation; vapour is produced rapidly to fill the partial vacuum. In this range of pressure, the heat is given by radiation or condensation; the convection effect is negligible, due to low air density.

The last stage of freeze drying is secondary drying. In this phase unfrozen water molecules are cleared. The temperature is raised greater than the primary-drying phase. This is carried out to break any physico-chemical interactions that have formed between the frozen material and the water molecules. After completing this process, the vacuum is usually broken with an inert gas (nitrogen) to make sure there are no reactions present between the gas and the materials. The technique freeze-drying is used in pharmaceutical, food industries, technological industry ect. (En. wikipedia.org, 2019), (Pearson 2019).

In the industry aspirin is made in larger quantities and is made through a batch process. A batch process is a manufacturing process that is a short process. It is done in small quantities and of limited number. It does not carry on going once it has started and is the opposite of a continuous process. Aspirin can be tested through two techniques, the measurement of melting point and the appearance of crystals. (Pearson 2019)

Testing purity of an organic substance through the appearance of crystals.

When testing the appearance of crystals, the individual should be able to recall what the product is to look like. According to that, many questions are to be answered to test whether the organic substance is pure or impure. For example, questions that are likely to be asked are, whether the organic substance should be a solid? Should it be colourless or not? Is it crystalline? And what shape should the crystal be? If the answers are different to how the product is meant to look like, the substance would be considered as impure. (Pearson 2019)

## Testing purity of an organic substance through the measurement of melting point

A substance can be identified if it is pure or impure through comparing the range in melting points. The melting point is the temperature at which the substance exists in both states, liquid and solid. The measurement of melting point is carried out using the equipment a Galen-Kamp melting point apparatus. This determines the melting point of a substance through observing the sample. The measurement of melting is first taken when the first indication of melting is shown till the disappearance of the substance or organic solid. Pure solids melt at an exact temperature, this is usually within 1 or 2 degrees Celsius of the published value, which can be found on the internet or through books. However, impure solids melt over a large range in temperature. The melting point of a substance may occur due to the addition of another element. Inorganic solids melt at extremely high temperatures; therefore, they have to be tested in industries and not school laboratories. This technique of testing the purity of an organic substance allows chemists to get accurate indications of the purity of organic and inorganic substances. (Pearson 2019)

## Preparation and testing purity of an organic liquid and drawing conclusions

### Equipment

- Condenser - Used to change the state of a substance from a gas to a liquid
- Clamp and stand - Used to hold equipment firm in place
- Isomantle - Used to heat substances

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- Water source – Needed for the experiment
- Round-bottom flask – Used for chemical reactions and reflux's
- Tripod – Used to put beakers and bowls on to heat substances over a Bunsen burner
- Bunsen burner – Used to heat substances
- Heat proof mat – Used to put beneath the Bunsen burner to protect the surface
- 2-Hydrobenzoic acid, salicylic acid, phosphoric acid – needed to make aspirin
- Spatula – Used as a spoon to scoop solids out
- Weighing boat – Used as a container
- Scale – Used to weigh the amount of a substance.
- Measuring cylinder – used to measure solutions accurately
- Fume cupboard – Used for ventilation to protect from exposure of chemicals
- Beakers – Needed for the practical
- Ice bath – Used to cool solids and liquids
- Pipette – Used to measure solutions precisely
- Vacuum – To draw in pressure
- Glass rod – Used to mix substances or solutions together
- Buchner filter – To filter products easily

#### Method for manufacture of aspirin

1. The apparatus for the practical was set up.

2. A round bottom flask was weighed and recorded. Using a spatula 2g of 2-hydroxybenzoic acid (salicylic acid) was taken out into the round bottom flask and weighed.
3. 4cm<sup>3</sup> of ethanoic anhydride and a few drops of phosphoric acid was added to the round bottom flask. This process was done in a fume cupboard.
4. The flask was clamped and the condenser was attached. Then the flow of water was checked, ensuring the quick fit apparatus was watertight.
5. The isomantle was turned on and adjusted to a low heat. The round bottom flask was held just above the compartment of the isomantle. This mixture was then swirled and further heated for ten minutes to ensure all solid particles had completely dissolved.
6. Next the mixture was left to cool before dismantling the condenser from the apparatus.
7. To the solution, 5cm<sup>3</sup> of cold water from the tap was added. The condenser was then put into an ice bath, until all the solid had formed.

#### Method for recrystallisation method

1. A double filter paper was wetted slightly using tap water and then the product was then filtered using a Buchner filter. This was then washed with cold water.
2. The product was then recrystallised using the smallest amount of water.
3. This was then left in the sun overnight to dry completely.

#### Determining the yield of aspirin for testing the purity of organic solid.

Results:

Mass of bowl and aspirin = 76.64g

Mass of bowl = 74.86g

Actual mass of aspirin = 1.78g  $\approx$

1.8g

Melting point of aspirin = 135 degrees Celsius

Amount of aspirin used in the practical = 2g

Theoretical yield: = Number of moles(m) = Mass Mr

of Hydroxyl benzoic acid

No of moles = 2 g 138

= 0.014gmol<sup>-1</sup>

Mole ratio = 1:1

Percentage yield = Actual yield Theoretical yield X 100 = 1.82 X 100 = 90  
%

Atom economy = Mr of desired product Mr of all reactants X 100

M<sub>r</sub> of desired product = M<sub>r</sub> of Aspirin

M<sub>r</sub> of C<sub>6</sub>H<sub>4</sub>COOHCOOCH<sub>3</sub> = 180gmol<sup>-1</sup>



So, the atom economy =  $180 \div 240 \times 100 = 75 \%$

$M_r$  of all reactants =  $M_r$  of 2-hydrobenzoic acid +  $M_r$  of ethanoic anhydride.

$M_r$  of  $C_6H_4OHCOOH + C_2O_3CH_3CH_3$

(C= 12, H= 1, O= 16)

$138\text{g mol}^{-1} + 102\text{g mol}^{-1} = 240\text{g mol}^{-1}$

### Melting point determination for purity of aspirin

Aim: In this practical, reacting acetic anhydride with salicylic acid in the presence of phosphoric acid will synthesise aspirin. After synthesis, the sample is purified via recrystallisation. The purity is analysed using the Siwoloboff method, which is checking purity by comparing melting points with the ones from the values taken from the experiment and literature values. The results will then produce a theoretical yield, in which a percentage yield will be shown and an atom economy. Depending on the temperature melting point of the samples, it will show if it is an impure or pure substance.

(Magbri. M 2014)

Equipment:

- Capillary tube – This is used to draw in solutions or solids. Ensure this is sealed off at one end.
- Aspirin from practical – This is needed for the practical, packet aspirin, aspirin made from previous year.

- Pestle and mortar - Used to grind materials, in this practical aspirin is grinded to powder form.
- Galen-kamp melting point apparatus - Used o determine the melting point of a substance.
- Ruler - Used to measure lengths.
- Thermometer - Used to measure the temperature.

Health and safety:

Goggles - This was worn to protect the eye(s) from any substances from entering the eye.

Gloves - This was worn to protect the skin on the hands.

Lab coat - This was worn to protect clothing.

Method:

1. Aspirin from the practical was grinded using a pestle and mortar until it had formed into a powder.
2. Using a sealed capillary tube, aspirin powder was drawn in. This was done through scooping aspirin into the opening of the capillary tube and then tapping the bottom of the capillary tube onto the surface vertically, so that the aspirin would reach the bottom of the capillary tube.
3. This was done several times until 3-4cm of aspirin had reached the bottom of the capillary tube, this was measured using a ruler.

4. Steps 2-4 were repeated, using one tablet from a packet aspirin brought from a local pharmacy and a small amount of aspirin taken from the practical carried out from the previous year.
5. The capillary tubes were then put into the oven block of the Galenkamp melting point apparatus next to the thermometer.
6. When the sample began to melt the temperature was noted down and when the sample had melted completely, the temperature was also written down. This step was carried out for all the samples.

Results:

Range

of

melting

point

Aspirin points

(degrees

Celsius

)

Packet 135-

aspirin 150

Practical

125-

aspirin

151

aspirin

Previo

us

year's 120-

practic 151

al

aspirin

Conclusion:

From the table, it shows that the packet aspirin began to melt at  $135^{\circ}\text{C}$  and completely dissolved at  $150^{\circ}\text{C}$ , hence it had a range of 15. The practical aspirin started to melt at  $125^{\circ}\text{C}$  and had completely melted at  $151^{\circ}\text{C}$ , hence it had a range of  $26^{\circ}\text{C}$ . The aspirin from the previous year started to melt from  $120^{\circ}\text{C}$  and had completely melted at  $151^{\circ}\text{C}$ , hence it had a range of 31.

Analysis:

To identify the purity of a substance, whether it is pure or impure, can be carried out through comparing the melting points. If the range of the melting point is small, it shows that the substance is pure, however if the range of the melting point is large, then it shows that the substance is impure. From the table shown, the aspirin which had the lowest range in melting point was the purest of all aspirins. Therefore, the packet aspirin

from the local pharmacy was the purest. This is because it had the lowest range of 15 in comparison to the other ranges 26 and 31. The other two

substances, practical aspirin and previous year's aspirin had greater ranges in melting points in comparison to the packet aspirin, this implies that they had the lowest purity.

Evaluation:

-Labelling the capillary tubes of what type of aspirin is contained, will make the process clearer and stop confusion of which capillary tubes contain which aspirin.

- To further test the purity of aspirin, TLC method can be used.
- During the transfer period from one product to another, the amount of product wasted is a common error and may affect the purity of the product.
- Another error is during the squeezing process, a possibility of pressing too hard using pressure, may rip or tear the filter, therefore it may need to be discarded and start again. (Odinity 2019).
- Additionally, an error is spillage of the solution or product due to human error. In an environment there are dust particles and other conditions that could alter results of the practical. (Odinity 2019).

#### Comparison between the laboratory and industrial manufacture and testing of an organic solid

In an industry, the process of manufacturing for aspirin is done through a batch process. This is when the reactants are added to the reactor and the reaction is started again from the beginning, it does not carry on in a loop or cycle. This progress is monitored carefully and the reactor is emptied at the <https://assignbuster.com/industrial-manufacture-and-testing-of-an-organic-solid/>

end of the reaction. The product mixture then continues onto the separation and purification stages. This process is ideal for small quantities of product.

In the laboratory, the process of manufacturing is done through a continuous process. In a continuous process reactants flow into the reactor at one end and products flow out of the other end, this process then continues throughout, it does not stop. This process is ideal for large quantities of product.

Percentage yield:

In a practical carried out within a laboratory the average percentage yield is 20.77%, whereas in a industry, when the practical of aspirin is carried out it results in an average percentage yield of 79.22%. It is difficult to obtain a percent yield of 100% because products can react to produce the reactants. (Magbri. M 2014).

Effectiveness of the process: During the experiment one of the most significant and obvious observations was the inefficiency of small-scale synthesis reactions in the manufacture and production of aspirin in a laboratory. The overall evaluation, is that manufacture and production of aspirin is more valuable and effective with a yield of 90% or more within an industry as more product is produced due to large-scale commercial applications and specialised tools.

A catalyst is used within aspirin to speed up the reaction without being used up, whilst producing an appreciable rate. To induce crystallisation, both

methods of the use of scratching the side of the beaker and Phosphoric acid in the synthesis part of the experiment and can be observed. (Odinity 2019)

Management of waste: Global warming has many contributing factors such as wasted energy that is caused by pollution. This wasted energy can be highly costly in the chemical industry. In order to be more cost effective manufacturing plants will look at ways to decrease this energy costs, such as, use previous waste to generate energy, for local housing sell excess energy for supply, switch to alternative methods that require less energy ; the heat energy that is produced from exothermic reactions and re-using it. (St a and st b s Lanark sch uk 2019)

Health and safety: Avoid contact with eyes and skin of the chemicals, acetic anhydride and phosphoric acid, as these are both corrosive and acidic. If however you come into contact with any of these chemicals, wash the area with cold water and antibacterial soap. Avoid inhaling any of the vapours as Acetic anhydride it an irritant. As the aspirin made is not pure enough to use as a drug, ensure it is disposed safety and correctly. (Webassign net 2019)

Cost: Manufacturing costs can be divided into capital costs, variable costs and fixed costs.

Capital costs - These are large quantities of money needed to set up a process, this includes, constructing, property, infrastructures and research and development.

Variable costs – These are in relation to chemical process involved, which are not incurred if production stops. For example, raw materials, product distribution, energy and overheads and effluent treatment/disposal.

Fixed costs – In relation to volume of production. Whether the production is high or low, these costs are still incurred. The effect on the selling price of the product decreases as a scale of production increases. For example, labour costs, , land rental and sales expenses depreciation of plant and plant maintenance. (St a and st b s Lanark sch uk 2019)

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