

Preparation and recrystallisation of aspirin biology essay



**ASSIGN
BUSTER**

The objective of this experiment is to enable us to understand and conduct the synthesis of aspirin, understand the skills of recrystallisation and the technique of melting point determination. The main procedures are preparation of aspirin, recrystallisation of aspirin and lastly determining the melting point of the aspirin. For preparation of Aspirin, acetic anhydride is added to the measured amount of salicylic acid. Sulphuric acid is added and heated for a short period to complete reaction. Water is added once removed from heat with addition of cold water and suction filtration is carried out. As for recrystallisation of aspirin, collected crude product prepared in preparation of aspirin which is impure is dissolved in ethanol and hot distilled water is added to the solution. The watch glass together with the filter paper was weighed and the weighed filter paper was used to carry out the suction filtration. Moreover, the crystals were placed on the watch glass after drying and its weight was calculated. Then, determine the melting point of aspirin using necessary apparatus. The percent yield was about 76.7% whereas the temperature range is between 134.2 to 136.1 °C. The results are within the expected range and I would conclude our experiment was a success.

Felix Hoffman who is a German chemist formed aspirin in 1897 by looking into French Chemistry Charles Gerhardt's experiment and rediscovered the acetylsalicylic acid and produced stable state of aspirin (The Great Idea Finder 2006). Salicylic acid was extracted from parts of willow trees (Dermaxime 2011).

Aspirin is a derivative of salicylic acid that is a mild agent that reduces pain by relieving headache and muscle and joint aches (History of aspirin 2011).

Aspirin is also used to treat mild to moderate pain from conditions such as <https://assignbuster.com/preparation-and-recrystallisation-of-aspirin-biology-essay/>

muscle aches, toothaches and headaches. Aspirin works in our body by blocking certain natural substances in your body to reduce pain and swelling (Aspirin 2011). Esterification is a chemical reaction that is used to make esters in which the Carboxylic acid combines with an alcohol to form an ester [CH₃COOC₂H₅] in the presence of a catalyst which is usually concentrated sulphuric acid. It is reversible reaction and they produce sweet smelling products. Esters are widely used in fragrance and flavour industry (Esterification reaction 2010). Aspirin has to be recrystallised several times to obtain purity. The properties that allow this to take place is its bulky chemical group, solubility in water and its strong intermolecular forces between the aspirin molecules that allows it to make up the solid substance.

Applications

Aspirin can be used in various medical uses such as relieving mild aches and pains. It can also be used for a little complex treatment such as prevention of blood clots and prevent heart attack and stroke (Aspirin and Heart attacks 2005). However, there are side effects such as internal bleeding to the stomach and other internal organs and also cause people to experience asthma attacks and undesirable swellings (Vitamin Diary, Aspirin 2010).

Theory

Esterification Reaction

Figure 1: Esterification Reaction (Preparation of Aspirin 1996)

Shown in figure 1, is the esterification process. To form aspirin, Salicylic acid is reacted with acetic anhydride using sulphuric acid as a catalyst to produce <https://assignbuster.com/preparation-and-recrystallisation-of-aspirin-biology-essay/>

Aspirin and Acetic acid. Sulphuric acid is used in this experiment as a catalyst to increase the rate of the reaction without being consumed in the process.

Ethanoic Anhydride

There are three reasons why ethanoic anhydride is used rather than other reagents such as ethanoyl chloride even though they yield aspirin. The reasons are, ethanoic anhydride is cheaper to be purchased compared to ethanoyl chloride. It is also safer to use as it is less corrosive and does not readily hydrolyse in water unlike the other reagent. Moreover, it does not produce dangerous fumes of hydrogen chloride like ethanoyl chloride (Chemguide 2004).

Procedure

Preparation of Aspirin

Firstly, approximately, 2.4g of salicylic acid was weighed into a dry 100ml conical flask and recorded. 6ml of acetic anhydride is added into the flask containing the salicylic acid in the fumehood. Then, 3-4 drops of concentrated sulphuric acid is added to the mixture and swirled. The mixture was also heated in the water bath for 10 to 15 minutes for the completing of reactions. After removing the flask from the water bath, 1ml of distilled water is added using a dropper to decompose the excess acetic anhydride present in the solution. 40ml of cold water is added and is stirred and rubbed using a stirring rod to induce crystallisation. After suction filtration is carried out the crude product is then collected.

Re-crystallization of Aspirin

An approximate 30ml of hot water is added to the solution and the crude product is dissolved in a 100ml conical flask with approximately 5ml of ethanol added to it and warmed on a hot plate. As there is solid separating out, the solution will be warmed till it is completely dissolved in the solution and was then allowed to cool. Then, a clean, dry watch glass with 2 filter papers was weighed and recorded. During the suction filtration of the solution, the recrystallised product is collected to the filter paper and was transferred to the watch glass. This was then put into the oven at 100oC for 15 – 20 minutes. Crystals together with the filter paper and the watch glass was then placed into desiccator for 5 to 10 minutes for all the liquid present to be drained. The dried crystals together with the filter papers and watch glass was weighed and recorded. The weight of dried, recrystallised aspirin is then calculated. The expected yield of aspirin was calculated from the amount of aspirin used and the percentage yield of the dried, recrystallised aspirin was calculated.

Melting point determination of aspirin

The aspirin was packed into the small capillary tubes and put into the optimeelt melting apparatus to determine the melting temperature of Aspirin.

Results

Mass

Mass of salicylic acid weighed (a) = 2. 40g

Mass of filter paper & watch glass (b) = 32.96g

Mass of dried, recrystallised aspirin, filter paper & watch glass (c) = 34.41g

Mass of dried, recrystallised aspirin (d) is calculated by taking the mass of dried, recrystallised aspirin, filter paper and watch glass (c) which is 34.41g and subtract away the mass of filter paper and watch glass (b) which is 32.96g.

$$(d) = (c) - (b)$$

$$= 34.41\text{g} - 32.96\text{g}$$

$$= 1.45\text{g}$$

Therefore the mass of dried, recrystallised aspirin is 1.45g.

Percent Yield

To obtain the percent yield of aspirin, we have to find the moles of salicylic acid used first by taking the mass of the salicylic acid which is 2.40g and divide by the mr of salicylic acid.

Number of moles of salicylic acid used (e) = mass/mr

$$= 2.40/138$$

$$= 0.017391 \text{ mol}$$

(mol wt of salicylic acid = 138)

Therefore the expected number of moles of aspirin (f) = 0.017391 mol

To calculate the expected mass of aspirin, we have to take the expected moles (f) which is 0.017391 mol multiply by the mr of aspirin 180.

$$\text{Expected mass of aspirin (g)} = 0.01739 \times 180 = 3.1302 \text{ g}$$

(mol wt = 180)

Then the percent yield can be calculated by taking the mass of dried recrystallised aspirin (d) which is 1.45g and divide by the expected mass of aspirin 3.1302g multiply by 100%.

$$\text{Percent yield} = (d) / (g) \times 100\%$$

$$= 46.3\%$$

4.3 Melting Point

Temperature range = 134.2 °C to 136.1 °C

4.4 Appearance

White, thin, flaky crystals formed.

Discussion

My Results

According to my experiment and the results, I conclude that my end product is not really pure. This can be seen from the calculation made based on my experiment using this formula,

Percent yield = (mass of dried recrystallised aspirin / expected mass of aspirin) x 100%

Percent yield is the amount of substance we have obtained in total in the experiment. The experimental yield percentage is different from the theoretical percentage is because there is loss of product often occurring during the isolation and purification steps (The Synthesis of Aspirin 2010). The percent yield of the aspirin obtained from the experiment is 46.3% yield. The higher the yield percentage, the higher the purity of the aspirin will be. Therefore, according to the results, the aspirin obtained is relatively impure. However, the low percent yield can also mean that the reactant has not reacted completely or the reaction is not complete. However there is also another possibility for the lower percent yield value. It is the addition of water when carrying out suction filtration. As we have to wash down the crystals before we carry out the suction filtration, some crystals might have dissolved. Hence, the amount of water we use to wash down the crystals during suction filtration might have affected the percent yield too.

The aspirin crystals are packed into the small capillary tubes and make sure they are all compressed without air gaps. Then they are placed into the melting apparatus. The melting temperature range of aspirin according to my experiment is between 134.2 °C to 136.1 °C. The theoretical melting temperature is 140 °C. Since the range is near the theoretical value, this shows that the aspirin obtained is quite pure and hence contained fewer impurities.

From both the calculations, I can evaluate that the aspirin is relatively pure to a however due to some experimental errors or improper techniques

<https://assignbuster.com/preparation-and-recrystallisation-of-aspirin-biology-essay/>

practiced, the percent yield is not up to expectation and moreover the incompleteness of reaction might also be one of the reasons for the results obtained in this experiment.

Experimental errors

There were some experimental errors that have caused variation in my results compared to the theoretical solutions.

Firstly, after obtaining the crude product from the first suction filtration, we had to transfer it to the conical flask to carry out recrystallisation. During this process, there were some crystals that got blown away by the wind and some crystals poured on the desk too. Hence this might have affected the percent yield too.

Therefore, I had learnt that all wind sources must be switched off and be kept away from when carrying out this process to ensure accuracy in results.

Secondly, once we have dissolved and during the second round suction filtration in an attempt to obtain the pure aspirin, we forgot to use 2 filter papers but instead used only one filter paper on the Buchner funnel. Hence, due to the pressure, the filter paper tore and our crude product entered the filter flask that was containing the impurities and other liquid. Therefore we had to suction filtrate the whole mixture in the filter flask and hence, this might have led to the presence of more impurities or loss in product. This might have affected the results.

Figure 2: Apparatus of suction filtration (Chemistry 104: Synthesis of Aspirin 2010)

<https://assignbuster.com/preparation-and-recrystallisation-of-aspirin-biology-essay/>

Therefore, from this experiment I learn that I must be more alert and careful when I carry out suction filtration to avoid unnecessary hassle and inaccuracy of results and calculation.

Conclusion

From this experiment, I have learnt how to carry out suction filtration in the right way and to be cautious at all time when handling chemicals and so on. The major experimental findings are that, accuracy and attentiveness is very important in this experiment to obtain aspirin that is pure. However, there will be some environmental effects that will still affect the experiment to a small extent.

Finally the objective of the experiment is met and the results were acceptable as it is quite accurate.

Recommendations

To improve on accuracy of results, we have to take into concern environmental factors such as the fan in the lab. It could affect the experimental findings as it blows away tiny light weight crystals while transferring them from one place to another. This would lead to loss of recrystallised crystals and cause variation in our calculations.

When carrying out the suction filtration, it is strongly advisable to use 2 or more filter papers to prevent the tearing of the filter papers during the suction filtration due to the pressure. This would lead to the crystals to be in contact with lots of water and cause the crystals to dissolve. This might also cause inaccuracy to results. Moreover, when transferring the crystals into the <https://assignbuster.com/preparation-and-recrystallisation-of-aspirin-biology-essay/>

Buchner flask before suction filtration, cold water is the most suitable to wash down the crystals as it minimises dissolving of crystals unlike fresh new solvents.

All the chemicals must be handled in the fumehood to prevent any corrosive chemicals such as salicylic acid, acetic anhydride and sulphuric acid to be in contact with our body. This is so as these chemicals can cause irritation to body parts such as eyes and skin and can also cause bad burns.