Synthesis and recrystallization of aspirin | lab report

Synopsis

The objective of this experiment is to enable us to conduct the synthesis of aspirin, reinforce the skills of recrystallisation and reinforce the technique of melting point determination. It is carried out to form ester from an acid and an alcohol. 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. Once solid dissolved, weigh the watch glass and filter paper, use the filter paper to carry out suction filtration, place crystals on watch glass, weigh the dried crystal and calculate the weight of the aspirin. 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 ÌŠÌŠC.

Introduction

Felix Hoffmann, a German chemist, produced a stable form of acetylsalicylic acid, more commonly known as aspirin; in 1897. Aspirin is a derivative of salicylic acid that is a mild, nonnarcotic analgesic useful in the relief of headache and muscle and joint aches.

Esterification is a chemical reaction used for making esters. The reaction in which a Carboxylic acid combines with an alcohol in the presence of a

catalyst (commonly concentrated sulphuric acid) to form an ester [CH3COOC2H5] is called Esterification reaction. It is reversible reaction and they product sweet smelling products. Esters are widely used in fragrance and flavour industry. . It is also useful in organic chemistry for the test of alcohols and carboxylic acid. It is used in polymer industry. Esterification reaction is used in the manufacture of paints, varnishes, lacquers, medicines, dyes, soaps and synthetic rubber.

Theory

Aspirin is a drug that is usually used to relieve minor aches and pain and other medical uses such as anti-inflammatory medication. Aspirin is an ester that has high molecular weight and it not soluble in water hence the solid can be separated by crystallization process. Synthesis of Aspirin is known as esterification.

Acetic anhydride is uses as it is cheap and forms a by-product, acetic acid which is not corrosive and can be recovered to make more acetic anhydride unlike other acetylating agents that also can be used.

All addition of chemicals to aspirin is done in the fumehood. Fumehood is a local ventilation device that is designed to limit exposure to hazardous or noxious fumes, vapours and dust. A fume hood is a large piece of equipment five enclosed sides of a work area, the bottom of which is most commonly located at a standing work height.

The three main purpose of the fumehood are as follows:

1. Protect the user carrying out the experiment

2. Protect the product and experiment from undesired reactions.
3. Protect the environment from emission of harmful products.

In the results, we calculate the percent yield and the melting temperature. Percent yield is calculated by (Mass of dried, recrystallised aspirin) ÷ (Expected mass of aspirin). In this experiment, the percent yield is 76. 7% yield which shows that it is almost fully pure and contains lesser impurities. The temperature is set to 100 ÌŠC and then gradually heated up to determine the melting point. The theory melting point is 140 ÌŠC and the experiment melting point range is 134. 2 ÌŠC to 136. 1 ÌŠC.

Procedure

Preparation of Aspirin

2. 4g of salicylic acid is measured in a 100ml conical flask and is recorded.

6mL of acetic anhydride is added to the salicylic acid to the flask in the fumehood.

3 to 4 drops of conc. Sulphuric acid is added to the mixture and is swirled. Mixture is heated for 10 to 15 min to complete reactions.

Suction filtration is carried out and the collected crude product after washing it a little with cold water.

Additional 40mL of cold water is added. Stir and rub mixture to induce crystallisation.

After removing from heat, 1 mL of distilled water is cautiously added to the mixture while it is hot to decompose the excess acetic anhydride.

Recrystallisation of Aspirin

Crude product collected was dissolved in approx 5mL of ethanol in a 100mL conical flask. Warmed.

Approx 30mL of hot distilled water is added to the solution. If solid separates, warm till solid dissolved completely.

Solution allowed to cool down.

The dried crystal is weighed together with the filter paper and watch glass. Weight is recorded.

Suction filtration is carried out to obtain the recrystallised product using the weighed filter paper.

A clean and dry watch glass is measured together with a filter paper and the weight is measured and recorded.

Weight of the dried, recrystallised aspirin, the expected yield of aspirin from the amount of salicylic acid, the percentage yield of dried, recrystalllised aspirin is measured.

Melting point is determined by using the optimelt melting apparatus.

Results and Calculations

Preparation and recrystallisation of Aspirin

Mass

Mass of salicylic acid (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) = (c) – (b)

= 34. 41 – 32. 96

= 1. 45g

Percent yield

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

= 2. 40/138

= 0. 017391 mol

(mol wt of salicylic acid = 138)

Expected number of moles of aspirin (f) = 0. 010507 mol

Expected mass of aspirin (g) = 0. 01739 x 180 = 3. 1302 g

(mol wt = 180)

Percent yield = (d) / (g) Ã- 100%

= 46. 3%

Melting Point

Temperature range = 134. 2 ÌŠC to 136. 1 ÌŠC

Appearance

White, thin, flaky crystals

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 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 percent yield of the aspirin obtained from my experiment is 46. 3% yield. The higher the yield percentage, the higher the purity of the aspirin will be. Therefore, according to my 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 ÌŠC to 136. 1 ÌŠC. The theoretical melting

temperature is 140 ÌŠC. Since the values are quite near, this shows that the aspirin we obtained is quite pure and hence contained less impurities.

From both the calculations, I can evaluate that the aspirin is pure to a large extent however due to some errors or improper techniques; the percent yield is not up to expectation and incomplete reactions might also be one of the reasons.

Experimental Errors

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

Firstly, after the 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 source 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 attempt to obtain the pure aspirin, we forgot to use 2 filter papers but instead use only one 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 presence of more impurities or lose in product. This might have affected the results.

Therefore, from this I learn that I must be more alert 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.