

Acetylsalicylic acid synthesis and purity test



Aspirin, also known as acetylsalicylic acid, dates back to 1897, when it was isolated by Felix Hoffmann, a chemist with a German company Bayer.

Aspirin is a salicylate drug, often used as an analgesic to relieve minor aches and pains, as an antipyretic to reduce fever, and as an anti-inflammatory medication (1). Acetylsalicylic acid is used in various conditions such as lower back and neck pain, the flu, common cold, burns, menstrual pain, headache, migraines, osteoarthritis, rheumatoid arthritis, sprains and strains, nerve pain, toothache, muscle pain, bursitis (inflammation of a bursa, a fluid-filled sac located around joints and near the bones), and following surgical and dental procedures (2).

Timeline of Aspirin

400 BC

Hippocrates was left historical records of pain relief treatments, including the use of powder made from the bark and leaves of the willow tree to help heal headaches, pains and fevers (3).

1763

Reverend Edward Stone experimented by gathering and drying a pound of willow bark and creating a powder which he gave to about fifty persons: it was consistently found to be a ' powerful astringent and very efficacious in curing agues and intermitting disorders'. He had discovered salicylic acid, the active ingredient in aspirin (4).

1829

Willow's active chemical constituent, Salicin, was identified in 1829 by the French pharmacist H. Leroux (5).

1838

Italian chemist Raffaele Piria, isolated Salicin, the active compound in the bark for the first time (6).

1853

Charles Gerhardt, a French chemist, mixed salicylic acid with sodium and acetyl chloride in 1853, creating acetylsalicylic anhydride. The procedure to make this compound was time-consuming and difficult, causing Gerhardt to abandon his project without marketing it (7).

1897

A German chemist named Felix Hoffmann, who worked for a German company called Bayer, rediscovered Gerhardt's formula. Felix Hoffmann made some of the formula and gave it to his father who was suffering from the pain of arthritis (3).

February 27th 1899

Aspirin was patented (3).

1914

At the beginning of the first world war in 1914 the Allies lost their source of aspirin and so they offered large prizes for anyone who could make aspirin (8). A young Australian chemist, George Nicholas, attempted his own

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production in a small pharmacy in Melbourne. The product was made by reacting salicylic acid with an acrid smelling liquid, acetic anhydride, while being heated. A pure aspirin was produced that more than met the purity requirements of the British Pharmacopoeia (9).

1930-1939

Bayer's patent to Aspirin runs out (10)(11) .

1974

Professor Peter Elwood conducts a trial into the effects of Aspirin on preventing heart attacks (12).

1982

English scientist Professor Sir John Vane and colleagues, Sune Bergström and Bengt Samuelsson win the Nobel prize for discovering the role of aspirin in inhibiting prostaglandin production (13).

1989

US researchers report preliminary study suggesting that aspirin may delay the onset of senile dementia (14).

1995

US researchers find that Aspirin may protect against Bowel cancer (14).

1997 +

Aspirin is now being used, or tested, to treat a number of conditions (1).

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Salicin

Salicin is closely related in chemical make-up to aspirin. When consumed, the acetalic ether bridge is broken down. The two parts of the molecule, glucose and the benzylic alcohol then are metabolized separately. By oxidizing the alcohol function the aromatic part finally is metabolized to salicylic acid.(15)

The Benzylic alcohol is circled in orange on the diagram. The Acetalic ether bridge is circled in red on the diagram.

Salicylaldehyde

In 1838, Raffaele Piria [an Italian chemist] then working at the Sorbonne in Paris, split salicin into a sugar and an aromatic component (salicylaldehyde) and converted the latter, by hydrolysis and oxidation, to an acid of crystallised colourless needles, which he named salicylic acid (3).

The Hydroxyl group is shown in red, as well as the aldehyde group.

Salicylic Acid

Salicylic acid has the formula $C_6H_4(OH)COOH$, where the OH group is ortho to the carboxyl group. It is also known as 2-hydroxybenzenecarboxylic acid. It is derived from the metabolism of salicin (16).

The Carboxylic acid group is circled in blue.

Aspirin (acetylsalicylic acid)

The ester group in the final product Aspirin is circled in red.

Aim 1: To Synthesise Aspirin

My method of synthesising Aspirin involves the reaction between Salicylic Acid and Acetic Anhydride. This is viewed as one of the most popular methods of synthesising Aspirin, as the reagents are available for use within a school laboratory, and the conditions required can be set up with the use of basic heating equipment such as a Bunsen burner and different water baths.

The synthesis of aspirin is classified as an esterification reaction. Salicylic acid is treated with acetic anhydride, an acid derivative, causing a chemical reaction that turns salicylic acid's hydroxyl group into an ester group (R-OH + R'-OCOCH₃). This process yields aspirin and acetic acid, which is considered a by-product of this reaction. Small amounts of Sulfuric acid (and occasionally phosphoric acid) are almost always used as a catalyst (17).

Synthesising Aspirin from Salicylic Acid is the preferred way of producing Aspirin, as synthesising it directly from the Willow bark would prove very impractical and time consuming, as well as not providing me with the means to compare two commercial methods of making Aspirin.

Hydrolysis

Raffaele Piria, the Italian chemist who successfully isolated Salicylaldehyde from Salicin, did so by the process of hydrolysis and oxidation.

Hydrolysis is defined as the chemical process of decomposition involving the splitting of a bond and the addition of the hydrogen cation and the

hydroxide anion of water (18). In simple terms, it is the addition of water to split a compound. Here is an example of a hydrolysis reaction, where an Ester compound (Ethyl Ethanoate), is split by water in the presence of an acid catalyst, into ethanoic acid and ethanol. The reaction involves the splitting of the Ester bond (-COO) in Ethyl Ethanoate, to give the two compounds that the Ester was made from.

Hydrolysis is a key component of the conversion of Salicin to Salicylic acid. It involves the hydrolysis of Salicin to form the intermediate compound Salicyl Alcohol (Saligenin).

From the following diagrams, I have been able to conclude the reaction mechanism for the hydrolysis reaction. A molecule of water is added to the Salicin complex, which causes the splitting of the Ether and the formation of 2 separate compounds. The -OH from the water molecule joins to the Benzene ring, where the Oxygen atom from the Ether group was previously found in Salicin. The splitting of Salicin into two separate compounds is indicated by the line drawn across the Ether group.

The addition of the -OH group from the water results in the formation of Salicyl Alcohol, which is shown below. The -OH group which has been added is circled in red:

The other product formed from the hydrolysis reaction is glucose. The Hydrogen atom which has been added from the water to form the glucose molecule from the splitting of Salicin is circled in blue:

Glucose is the by-product of the reaction, and Salicyl Alcohol moves onto the next stage of the process, which is oxidation.

Oxidation

Oxidation is the loss of electrons from a compound, and is often indicated by the change in charge of an atom. A simple example is the formation of a Magnesium ion from magnesium metal during the reaction between Magnesium metal and oxygen to form Magnesium oxide.

The half equation is as follows:



We can see that the Mg has changed oxidation state, increasing to a charge of 2+, which shows it has lost two negatively charged electrons (e-).

The next stage of Piria's reaction involves the oxidation of Salicyl alcohol to form Salicylic Acid.

The Carbon atom circled, accepts an oxygen atom to form a Carboxylic acid group (-COOH), and is formed from a double covalent bond between the Carbon and oxygen atom. Accepting the oxygen atom, the Carbon atom loses electrons to the oxygen atom to form the double covalent bond, and the Carbon atom increases in charge and polarity to a more positive charge. This is because Oxygen is a more electronegative element, and attracts the electrons from the covalent bond more strongly than Carbon, which gives the Carbon atom a partial positive charge.

The colourless, organic crystalline compound produced is Salicylic Acid, and is the final product of this hydrolysis and oxidation react. However when it was used as a medicine, Salicylic acid was found to irritate the stomach lining and cause painful side effects in patients due to its relative acidity. Chemists tried to create a buffer to eliminate the painful irritation of the stomach lining due to the acidic nature of Salicylic Acid. In 1853 Charles Gerhardt, a French chemist, neutralised Salicylic acid by mixing it with sodium and acetyl chloride, creating acetylsalicylic anhydride.

The buffer produced was Sodium Salicylate, which was then reacted with acetyl Chloride, to produce Acetyl Salicylic Acid, the chemical name for Aspirin.

Nucleophilic Substitution Mechanism

The reaction between Salicylic acid and Ethanoic Anhydride/Acetyl Chloride is found as being a Nucleophilic Substitution reaction. A nucleophile is a chemical species that donates an electron pair to an electrophile to form a chemical bond in a reaction. All molecules or ions with a free pair of electrons are able to act as nucleophiles.

The nucleophile in this case is the Salicylic Acid, as it has a lone pair of electrons on the oxygen atom which is located in the hydroxyl group.

Ethanoic Anhydride is often used as the Ethanoylating agent because it is reactive but not too unpleasant or dangerous. A much more reactive Ethanoylating agent is Ethanoyl Chloride but this is toxic and hazardous to use because it is so reactive (1A).

Electronegativity

A basic principle which must be explained in order to understand the reaction mechanism is the fact that the oxygen atoms on the Ethanoic Anhydride are much more electronegative than the Carbon atoms.

Electronegativity is a measure of the strength of attraction between an atom and an electron pair. It is known as 'electron pulling power'.

We can therefore use differences in electronegativity to predict how polar atoms in a compound will be, allowing us to work out the reaction mechanism.

According to Pauling's electronegativity values, oxygen is a much more electronegative element than carbon. (Oxygen has a value of 3.4, compared to Carbon having a value of 2.6).

(19)

This gives the oxygen atoms present in Ethanoic Anhydride a partial negative charge, shown as δ^- , while the Carbon atoms have a partial positive charge, which is shown as δ^+ . This means that the Oxygen atom attracts electrons more strongly than the Carbon atom.

The Oxygen atom in the Phenol group in Salicylic acid is also δ^- . As well as this, the oxygen in the Phenol group has a lone pair of electrons due to the type of bonding it shows.

This lone pair induces attraction between the δ^- oxygen atom in Salicylic acid and the δ^+ Carbon group on Ethanoic Anhydride. The Salicylic Acid

therefore acts as a nucleophile, and the lone pair on the oxygen atom in the Phenol group of Salicylic acid attacks the δ^+ carbon atom in Ethanoic Anhydride.

This results in the substitution of a hydrogen atom from the phenol group, as well as the CH_3COO^- anion from the Ethanoic anhydride. These then bond, to form CH_3COOH , which is known as Ethanoic Acid, and is the waste product of the reaction.

The phenol group is therefore able to esterify with ethanoic anhydride to produce aspirin.

(20)

During the course of my investigation, I will compare the 2 methods used to synthesise Aspirin from Salicylic Acid. Both my methods involve the use of Ethanoic Anhydride, and follow the same reaction mechanism as shown above. However the differences between the methods are in the reagents and conditions used to synthesise Aspirin. Through my tests for purity, and percentage yield, as well as my comparison of each method with commercially produced Aspirin, I will be able to conclude which method is most efficient.

Catalysts

My methods involve the use of two different catalysts.

The definition of a catalyst is a substance which speeds up a chemical reaction by lowering the activation enthalpy, but can be recovered chemically unchanged at the end (1C).

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Due to the physical nature of the reagents, both the catalysis involved in the reactions are Homogeneous.

A homogeneous catalyst is one that is in the same physical state or phase as the reactants. In both methods of synthesising Aspirin, this is the case, as shown below:

Method

Reactants

Catalyst

1

Salicylic Acid and Ethanoic Anhydride (in solution)

Concentrated Sulfuric Acid (aq)

2

Salicylic Acid and Ethanoic Anhydride (in solution)

85% Concentrated Phosphoric Acid (aq)

As shown in the table, both reactants and catalyst are in solution, making the process Homogeneous Catalysis.

The catalyst is added to the solution of Salicylic acid and Ethanoic Anhydride in order to lower the activation enthalpy of the reaction, so in effect, a lower temperature is needed for the reaction to progress. This can be visually

described by an enthalpy profile, which shows the energy of the products and reactants along the y axis, and the progress of reaction along the x axis.

<http://www.chemguide.co.uk/physical/basicrates/catprofile.gif>

The Acid Catalysed acylation Reaction mechanism can be worked out based on the fixed mechanism involved in Esterification. Here are the steps:

Step 1: The proton from the acid attacks the carboxyl oxygen which in turn “pushes” the two electrons in one of the bonds “down”, (it delocalizes the electrons and “spreads them out” between the two Oxygen atoms).

Step 2: The delocalized electrons then, in the presence of the alcohol group, rearrange in such a manner as to create a temporary bond between the two reactants, forming an intermediate.

Step 3: The proton from the -OH group attacks the oxygen in the original -OH portion of the acid forming a positively charged oxygen atom. The Electrons holding the water molecule to the intermediate “flip down”, releasing this water, leaving the delocalized intermediate in the end of Step 3.

Step 4: In the final step, the proton added in Step 1 leaves and the electrons left behind flip down “closing” the double bond on the oxygen atom and leaving the ester product.

The mechanism for the homogeneous catalysis that takes place in the synthesis of Aspirin is shown below in skeletal formula, broken down into 6 steps:

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The formation of an intermediate in acid-based catalysis (as shown in step 2), is a characteristic feature of homogeneous catalysis. The enthalpy profile for the synthesis of Aspirin should look something similar to this:

(22)

As can be seen from the Enthalpy profile, there are two humps for the catalysed profile.

One is for each step of the reaction. The intermediate compound then breaks down to give the product and reform the catalyst (1D).

Aim 2: Purifying Aspirin using Recrystallisation

The sample of Aspirin which is directly obtained after vacuum filtration is very likely to contain impurities. These impurities will add to the mass of my sample of Aspirin, which will give me inaccurate results for percentage yield, as well as giving me inaccurate results when I test the purity of my Aspirin.

For these reasons, it is very important that I purify my sample of Aspirin immediately after synthesising it to remove impurities.

The different impurities that could be present in my sample of Aspirin cover a range of possibilities. For example, the sample could contain filter paper from the Hirsch funnel or drying. It could also have been contaminated from the use of lab equipment such as pipettes, beakers and funnels. As well as this, not all of the Salicylic Acid may have been converted to Aspirin, and so there may be some Salicylic acid in the Aspirin sample.

Another important factor that may be overlooked is the fact that throughout the process of synthesising Aspirin, acid is involved, often as well as high temperatures. Although this may not seem a concern, Aspirin can be easily hydrolysed back to Salicylic Acid and Acetic Acid. On their own, water and an ester react very slowly, but the process can be speeded up by catalysis (with acid or alkali) (2A). As in both my methods of synthesis, Acid is used (Sulfuric and Phosphoric). It is possible that Salicylic acid and Acetic Acid may be present in my final product as hydrolysis could have taken place (as shown below).

http://images.flatworldknowledge.com/averillfwk/averillfwk-eq14_005.jpg

(23)

The process of recrystallisation for purifying compounds is ideal because it removes insoluble impurities as well as soluble impurities.

Aim 3: To Identify my products as being Aspirin

It is essential that after purifying my products, I am able to accurately identify both as being Aspirin. If my products are not able to be identified as Aspirin, then it will render the rest of my investigation useless, because I will not be testing the purity of Aspirin. The main method of determining my product is by its melting point, which I will discuss in further detail later in this investigation.

However it is important to analyse the factors that may cause my product not to be Aspirin, and to do this I must look into Hydrolysis in more detail:

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I use acid catalysts in order to synthesise my Aspirin in both methods. When Aspirin is heated with acid in solution, it undergoes hydrolysis. The ester bond breaks and the two compounds join with an -H and an -OH group from the water, to form Salicylic Acid and Acetic Acid. There is the possibility that this may have taken place during my investigation due to human error. The mechanism for this acid based hydrolysis is shown below:

Mechanism of the Acid catalysed Hydrolysis of Esters

Step 1:

An acid/base reaction. Since there is a weak nucleophile and a weak electrophile, protonation of the ester carbonyl allows it to become more electrophilic. Here the acid is used, because of the Bronstead-Lowry Theory that an acid is a proton (H^+ ion) donor.

Step 2:

The lone pair on the O atom in the water molecule functions as the nucleophile and attacks the electrophilic C in the $C=O$, with the electrons moving towards the oxonium ion, creating the tetrahedral intermediate.

Step 3:

An acid/base reaction. The oxygen from the water molecule is deprotonated, and loses a H^+ ion.

Step 4:

Another acid/base reaction. The $-OCH_3$ group is lost, but first by protonation, and gains an H^+ ion.

Step 5:

Another acid/base reaction. De-protonation of the oxonium ion reveals the carbonyl in the carboxylic acid product and regenerates the acid catalyst.

It is important that I identify my product as Aspirin because if Hydrolysis takes place I may be left with traces of Acetic or Salicylic Acid in my product, which will give me inaccurate results for the rest of my investigation.

I will also use the melting point of Aspirin to determine the chemical nature of my products. Aspirin melts at a fixed temperature, and will therefore allow me to identify my product as Aspirin if it melts at the same temperature.

Aim 4 and 5: To Test the Purity of My Aspirin and compare my Aspirin samples and Methods.

There are a number of different methods of testing the purity of my Aspirin.

Testing the purity of my Aspirin shows how successful the recrystallisation method was at removing the impurities from solution.

The methods are as follows:

Thin Layer Chromatography: This technique is used to separate small quantities of organic compounds.

Titration with known samples of Sodium Hydroxide: This technique is referred to as an 'aspirin assay' and is used to compare my Aspirin from both methods, as well as with shop-bought Aspirin.

Iron (III) Chloride Test: This test is used to detect the presence of Salicylic acid, and therefore indicate an impure sample.

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Reaction with Sodium Bicarbonate: Aspirin will dissolve in Sodium Bicarbonate solution to produce Carbon Dioxide and sodium acetylsalicylate salt.

These 4 methods will allow me to test the purity of my Aspirin products. As well as this, I will be able to fulfil aim 5 of my investigation and compare the 2 Aspirin samples, therefore finding which method produces the purest sample of Aspirin.

Methods

Method 1: Synthesise Aspirin

Equipment and Chemicals

Fume cupboard

150cm³ conical flask

20cm³ measuring cylinders

Salicylic acid

Ethanoic anhydride

Concentrated Sulfuric(VI) acid

Glacial Ethanoic acid

Water bath containing crushed ice

Funnel

Procedure and Chemical Quantities

After zeroing the scales using a weighing boat, I weighed out 10.03g of Salicylic Acid.

I then measured out 20cm³ of Ethanoic Anhydride using a 150cm³ conical flask.

I carefully added the 10.00g of Salicylic Acid to the 20cm³ of Ethanoic Anhydride and swirled the solution in the flask. This took place in a fume cupboard.

Using a pipette, I then added 25 drops of Sulfuric (VI) acid to the flask, swirling the flask after every drop added. The acid acts as a catalyst.

I continued to swirl the flask until impure crystals of Aspirin began to form a 'crystalline mush'.

I then added 20cm³ of glacial ethanoic acid to the conical flask containing the mixture, which diluted the solution.

I then placed the flask in a cold water bath containing crushed ice to allow the crystals to form.

I then proceeded onto vacuum filtration.

Vacuum Filtration equipment

Buchner funnel

Filter paper

Clean solvent (e. g. Distilled Water)

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250cm³ volumetric flask

Tubing

Procedure

I connected the conical flask to a vacuum pump via the side arm. The pump creates a partial vacuum so that the filtrate gets pulled through quickly.

I dampened a piece of filter paper and placed it flat on the vacuum funnel, ensuring the whole area was covered.

I switched on the vacuum pump and carefully poured the Aspirin mixture in to be filtered.

I then disconnected the flask from the vacuum pump before turning it off. This avoided 'suck back'.

I then placed the filter paper containing my impure Aspirin carefully onto a watch glass and put it into an oven for 10 minutes set at a fixed temperature, to allow the crystals to dry.

Diagram of Vacuum Filtration

(24)

<http://upload.wikimedia.org/wikipedia/commons/thumb/d/da/Vacuum-filtration-diagram.png/623px-Vacuum-filtration-diagram.png>

Synthesising Aspirin: Method 2

1. I used a balance to weigh a 50 mL Erlenmeyer flask. I then placed 10.08g of salicylic acid in the flask and weigh again. In the fume

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cupboard, I then transferred 25.0 mL of acetic Anhydride from a burette into a 100ml flask, and added it to the flask containing the salicylic acid. I also added 5 drops of 85% phosphoric acid (catalyst) to the flask.

2. I clamped the flask in a beaker of tap water supported on a ring stand over a burner flame. I stirred the mixture until the salicylic acid had dissolved completely. The water was heated until boiling point, and then the flame was shut off. The flask was kept in the hot water bath for 10 more minutes.
3. While the flask was still in the water bath, I added 10 mL of distilled water to the flask to decompose any excess acetic anhydride.
4. After a minute, I removed the flask from the water bath and added 20 mL of distilled water. This Let the flask cool to room temperature. As the solution cooled, crystals of aspirin began to appear. The solution was cooled by placing the reaction flask in an ice bath.
5. I then weighed a watch glass and filter paper on the centigram balance.
6. I then proceeded to the vacuum filtration step as shown in the previous method.

Variations between two methods

The main differences between the two methods of synthesising Aspirin are in the catalyst involved.

Both catalysts are acid catalysts, as Method 1 involves Sulfuric Acid as the catalyst, and Method 2 involves Phosphoric Acid as the catalysts. These

catalysts may appear similar on the surface, as they both have a similar structure as shown below:

Sulfuric Acid Structure (25)http://www.globalwarmingart.com/images/9/93/Sulfuric_Acid_Molecule_Formula.png

Phosphoric Acid structure (26)http://upload.wikimedia.org/wikipedia/commons/thumb/2/29/Phosphoric_acid.svg/220px-Phosphoric_acid.svg.png

Although the structures appear the same, the most important factor in determining the effectiveness of a homogeneous catalyst such as an acid is to analyse its ability to transfer H⁺ ions in solution.

This is particularly important in the synthesis of Aspirin because H⁺ ions are added and removed from the intermediates and Phenol/Carboxylic acid groups as shown in my detailed explanation above.

The pH scale was devised at the beginning of the 20th century by a Danish Chemist called Soren Sorensen. He wanted a simple way to indicate how much acid or alkali was present in a solution. The 'p' in pH stands for potens, which is Latin for "power". So pH is measuring the power of Hydrogen ions in a solution, i. e. its concentration (2B).

pH is defined as:

$$\text{pH} = -\lg [\text{H}^+ (\text{aq})]$$

An acid which has a higher concentration of H^+ ions in solution is a stronger acid, and an acid that has a lower concentration of H^+ ions in solution is a weaker acid.

Stronger acids are typically used in dehydration and condensation reactions, as they are able to protonate other compounds.

In a dehydration the $-OH$ group becomes charged ($R-OH_2^+$) then it can leave (a double bond is formed) with the acid providing the extra H^+ for the alcohol. Then the resulting anion (HSO_4^- or $H_2PO_4^-$) can do the elimination reaction creating the double bond. If a stronger acid is used, the transfer of H^+ ions will be much faster, and the intermediate will be formed much more quickly, and the reaction is more likely to go to completion. For this reason, I will predict that the stronger acid catalyst is more likely to give the better yield and purity of Aspirin.

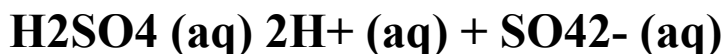
To come to a conclusion about which acid catalyst is the strongest, and therefore the strongest H^+ ion donor, the pH of both of the acids must be found. Through my research, I found that the pH of an acid solution can be calculated if the molarity of the acid is known.

To avoid any inaccurate values in my calculations, I will set the value of concentration at 0.1 mol/dm^3 for both Sulfuric and Phosphoric acid. This allows me to calculate a pH value for each acid which is irrespective of the concentration of the acid in solution, leaving the only variable as the chemical properties of each acid in donating H^+ ions.

My calculations are as follows:

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Sulfuric Acid



(The dissociation of Hydrogen ions in Sulfuric acid)

$$[H_2SO_4] = 0.1 \text{ M}$$

From the equation above, I can see that the molar ratio of Hydrogen ions dissociated compared to Sulfuric acid is 2: 1. This means that the concentration of Hydrogen ions will be 2x the concentration of Sulfuric acid, and 2 multiplied by 0.1 is equal to 0.2.

This allows me to put these values into the pH equation to calculate the value for pH.

$$\text{pH} = -\log [H^+]$$

$$\text{pH} = -\log [0.2]$$

$$\text{pH} = 0.699$$

Phosphoric Acid



(The dissociation of Hydrogen ions in Phosphoric Acid)

$$[H_3PO_4] = 0.1 \text{ M}$$

From the equation above, I can see that the molar ratio of Hydrogen ions dissociated compared to Phosphoric acid is 1: 3. This means that the

concentration of Hydrogen ions will be $\frac{1}{3}$ of the concentration of Phosphoric acid, and $\frac{1}{3} \times 0.1$ is equal to 0.03 recurring.

This allows me to put these values into the equation to calculate the value for pH.

$$\text{pH} = -\log [\text{H}^+]$$

$$\text{pH} = -\log [0.033]$$

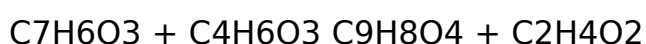
$$\text{pH} = 1.52$$

From these calculations, I am able to conclude that the pH of Sulfuric acid in 0.1 molar solution is stronger than the pH of Phosphoric acid, and is able to transfer more H^+ ions therefore, so theoretically should be the more effective catalyst in assisting Condensation reactions, and therefore should provide a better, purer yield of Aspirin. So Method 1 should theoretically be the most effective method.

Percentage Yield

Percentage Yield is used to see if a reaction is economically viable.

Using percentage yield will allow me to determine how effective the chosen method is at synthesising Aspirin. To calculate percentage yield for the reaction, I must use the overall equation for the reaction which begins with Salicylic Acid.



Salicylic Acid + Ethanoyl Chloride \rightarrow Aspirin + Acetic Acid

The only compounds I need to use for this calculation are highlighted in bold, and are Salicylic Acid and Aspirin.

The equation for Percentage yield is as follows:

$\% \text{ yield} = \frac{\text{Actual Mass of Product}}$

$\frac{\text{Theoretical Maximum mass of Product} \times 100}{\text{Theoretical Maximum mass of Product}}$

The Relative Molecular Mass of Salicylic acid is 138 and the Relative Molecular Mass of Aspirin is 180.

From this information, I can work out the Theoretic maximum yield of Aspirin.

In my investigation, I used 10.03g of Salicylic Acid while producing 11.12g of Aspirin.

The theoretical maximum yield of Aspirin from Salicylic Acid in terms of the quantities I used is calculated:

$180/138 \times 10.03 = 13.08\text{g of Aspirin}$

I use my calculated value for Theoretical yield with my value for my experimental (actual) yield to calculate percentage yield for Method 1:

$\% \text{ yield} = \frac{11.12}{13.08} \times 100$

$\% \text{ yield} = 85.02\%$

Method 1 has a high yield of Aspirin.

I will use the exact same process for method 2.

Using method 2, I used 10.08g of Salicylic Acid which produced 10.97g of Aspirin.

Theoretical Maximum yield = $180/138 \times 10.08 = 13.14\text{g}$ of Aspirin

% yield = $10.97/13.14 \times 100 = 83.48\%$

Method 2 has a slightly lower yield of Aspirin compared to method 1.

Note: There may still be impurities in the Aspirin, which mean that these results cannot be taken as final until the Aspirin has been purified. Impurities such as filter paper and lab contamination, as well as locked-in moisture may cause more mass than the Aspirin alone.

Method 2: Purifying the Aspirin- Recrystallisation

Recrystallisation is a technique used to purify solid crude organ