Preparation of Antifebrin

Safety of Chemicals

Phenylammonium Chloride ($C_6H_5NH_3Cl$)
- Highly Toxic
- Carcinogen (can cause cancer)
- Irritant to eyes and skin
- Harmful if inhaled, swallowed or touched

Sodium Ethanoate (CH_3COONa)
- May be harmful to skin
- Moderately toxic by ingestion
- Irritant to skin and eye

Ethanoic Anhydride ($(CH_3CO)_20$)
- Corrosive
- Harmful if swallowed
- Contact to eyes may cause irritation or burns

Antifebrin ($CH_3CONHC_6H_5$)
- Irritant
- Can cause a weak pulse and faintness if too much is inhaled or swallowed

Safety precautions

- Goggles will be worn throughout the experiment. This will protect my eyes against splashes of chemicals
- Safety gloves will also be worn throughout the experiment; this will protect my hands from the chemicals so it does not harm/damage my skin.
- A lab coat will be worn to protect my clothes and skin from the chemicals used.
- All glassware and containers will be moved into the middle of the table. This will ensure that they are not knocked of the table to cause a hazard. These containers will also be tightly sealed if not in use.
- I will use ethanoic anhydride in a chemical fume cupboard.
- While conducting my experiment I will keep all my equipment in my area and not take up to much space so that my neighbour can have enough space to conduct their experiment.
- When my experiment is finished I will wash my hand thoroughly with hand wash.

Observation

1. First step was to dissolve 1.0g of phenylammonium chloride in 30cm^3 of water. The phenylammonium chloride dissolved completely in the water, producing a fairly clear solution.
2. Second step was to prepare a solution of 6.0g of sodium ethanoate in 25cm^3 of water. The sodium ethanoate dissolved in the water leaving a few undissolved granules at the bottom of the flask. The solution was left fairly clear.

3. Third step was to add 2cm^3 of ethanoic anhydride to the solution of phenylammonium chloride. Before I began stirring a small solid formed, but as I started stirring it slowly dissolved leaving no precipitate and a clear solution.
4. Sodium ethanoate was added to the ethanoic anhydride and phenylammonium chloride solution. A white cloudy precipitate immediately formed, as I stirred the solution a thick solution with a lot of white/grey precipitate formed.
5. The precipitate formed was purified by filtering it under reduced pressure in a Buchner flask. This produce a white, shiny crystals, which however was still wet. Once dried the product became a dry powder form, crystalline.

Filtering

The product formed from the addition of ethanoic anhydride and solution phenylammonium chloride, needed to be filtered under reduced pressure and then washed with a little cold water. To filter the product a Buchner flask was used, the buchner funnel was attached to the flask and the other end to a fully flowing cold tap. As the tap was left running, the pressure in the flask was reduced. The pressure produced sucked the liquid in the hose barb, through to the conical flask below. In the hose barb was just the solid crystals.

Recrystallise

The crude sample of antifebrin needed to be recrystallised in step 5. To begin the sample was dissolved in a small amount of hot water so that that sample became saturated. After a couple minutes the solution was then allowed to cool in a bowl of ice, in which solid crystals formed. The solid crystals were then separated under reduced pressure, using the method described above.

Readings recorded

To ensure each reactant had a high degree of accuracy I repeated each measurement three times. When using the scales to measure reactants such as sodium ethanoate, I placed a small glass dish on the scales, and recalibrated the scales so that the weight of the dish was not included. After each weighing I wiped off and recalibrated the scales to make sure no small substances would disrupt the next reading.

Phenylammonium chloride

1.0g of Phenylammonium chloride was needed to dissolve into water. This was done by placing a dish on the scales and carefully added small amounts of phenylammonium chloride using a spoon. I repeated this three times, while recalibrating the scales after each weighing. The readings are as follows:

Reading 1/g	Reading 2/g	Reading 3/g
1.0	1.01	1.0

The mass of Phenylammonium chloride was therefore 1.0g, because only 2 significant figures were required.

Distilled water
For the first step 30cm^3 of distilled water was needed. I first pour a large amount of distilled water into a large beaker, then used a pipette to draw out water and emptied it out into a measuring cylinder and reading off the meniscus. I repeated this three times by using three different cylinders. The readings are as follows:

Reading 1/cm^3	Reading 2/cm^3	Reading 3/cm^3
30	30	30

The volume used was 30cm^3, of the third reading.

Another volume of 25cm^3 was needed to mix with sodium ethanoate. The same procedure to measure 30cm^3 of distilled water was used to measure 25cm^3

Reading 1/cm^3	Reading 2/cm^3	Reading 3/cm^3
25	25	25

The volume used was 25cm^3, of the third reading.

Sodium ethanoate
6.0g of sodium ethanoate was required. To achieve this I used scales to weigh out the measurement. I added tiny amounts at a time until the desired amount was reached. The results are as follows:

Reading 1/g	Reading 2/g	Reading 3/g
6.01	6.0	6.0

The mass used was 6.0g, from the third reading.

Ethanoic anhydride
2cm^3 of ethanoic anhydride was needed in this step, which at room temp was a liquid. I used the same procedure as that of water, but it occurred in a fume cupboard because of the corrosive nature of it. The readings are as follows:

Reading 1/cm^3	Reading 2/cm^3	Reading 3/cm^3
2	2	2

The volume used was 2cm^3.

Antifebrin
Once the final product, a white crystalline, was dried and purified I weighed it on the scales. I first weighed the empty specimen tube and then recalibrated it. I then added the product into the tube and recorded the weight. The weight recorded was:

Reading 1/g	Reading 2/g	Reading 3/g
0.44	0.44	0.44

The mass of the final product was 0.50g. To produce an accurate reading of Antifebrin, it must be thoroughly dried otherwise the mass of water will be included in the final weight. I dried off the antifebrin by dapping it with filter paper.

To measure the melting point required a small amount of dried Antifebrin, which was collected in a capillary tube. The tube required only a small amount which was tapped to allow the substance to settle at the bottom. The tube was feed into the electronic equipment, which would heat up to melt the substances in the tube therefore finding out the melting point. I was able to view the substances in the tube from the top of the equipment, to see we it had melted into a liquid. However it was difficult to pin point an exact melting point, the temperature at which the substance melted lie between 116^{o}C – 118^{o}C.

Percentage Yield

To find out the percentage yield the following formula will be used:

Actual mass / Expected mass x 100

Step 1: Find the moles of Phenylammonium chloride

Mass used in experiment = 1.0

Molar mass = 129.5

Number of mole = mass (1.0) / Mr (129.5)

= **7.722 x10^{-3}**

Step 2: Find the mass of Antifebrin

Molar mass of Antifebrin = 135

Mass = moles (7.722 x10^{-3}) x molar mass (135)

= **1.042 (3.dp)**

Step 4: Find the Percentage Yield

Percentage yield = actual mass / expected mass x100

= (0.44 / 1.042) x 100

= **42. 23 (2.dp)**

The percentage yield of this experiment is **42%.**