Determination of the percentage purity of Polopirin

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Aspect	D	DCP	CE
1			
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Total			

Aim: To Determine the amount of pure polopirin in a tablet. This should be done by means of back titration.

Design

Research Question: What mass of pure acetylsalicylic acid is present in the polopirin tablet?

Variables:

Table 1: Variables

Variables	
Independent	Mass of Tablet
Dependent	1. Mass of Acetylsalicylic acid in the tablet 2. Volume of HCL standard solution required to neutralise the excess of NaOH
Controlled	1. Number of Measurements 2. Amount of Phenolphthalein 3. Concentration of NaOH 4. Volume of NaOH 5. Concentration of HCl
Uncontrolled	1. Temperature 2. Pressure

Materials:

1) 50 cm^3 burette with precision of 0.1 cm^3
2) A 25 cm^3 Pipette with precision of 0.1 cm^3
3) 50 cm^3 Beaker
4) Dropper with Precision of 0.1 cm^3
5) A Weighing Scale with a Precision of 0.01 g
6) A Funnel
7) Mortar, Pestle

Chemicals:

1) Phenolphthalein
2) Polopirin (tablets)

3) 1.0 mol dm^{-3} solution of NaOH
4) 1.0 mol dm^{-3} solution of HCl

Procedure:

Back Titration was chosen to be used and performed during this practical, because aspirin is an insoluble, solid reagent, hence just back-titration can be performed.

A. Weigh the beaker.
B. Tare the Scale.
C. Crush the polopirin tablet with mortal and pestle and weigh out the sample.
D. Add approximately, 20 cm^3 of 1.0 mol dm^3 solution of NaOH using a pipette. Stir the mixture vigorously.
E. Add 3 drops of phenolphthalein using the dropper.
F. Install the burette on a stand and fill it with 1 mol dm^3 solution of HCl.
G. Titrate the excess base with HCl and observe the colour change.
H. Pour the acid until the colour of the solution turns from light pink/pinkish to transparent.
I. Record the volume of the acid required to neutralise the excess base.
J. Repeat the procedure 5 times.

Safety Rules:

All necessary safety precautions were undertaken. No lab rule was broken, everything went as planned.

The performers were wearing lab coats, rubber gloves, and protective glasses to ensure safety throughout the experiment.

Data Collection and Processing

Table 2 - Data Presentation with Standard Deviation Calculated

Mass of the beaker, [g] ± 0.01 g	Mass of the tablet in powder form , ± 0.01 g , m_{tablet}	Average mass of the tablet in powder form, [g] , m_{av}	Standard Deviation
29.71	1.443 g	1.442 g	0.011 g
	1.421 g		
	1.457 g		
	1.448 g		
	1.442 g		

Volume of HCl, [cm^3] ± 0.1 cm^3	Average Volume, V_{av}, cm^3	Standard Deviation ΔV_{av}, cm^3	Percentage Uncertainty, %
15.8	15.9	0.10	0.6 %
16.0			
15.7			
15.9			
15.9			

(Successive lines in the tables stand for each trail)

The standard deviation of the results was calculated using a formula:

$$S = \sqrt{\frac{\Sigma(X - \overline{X})^2}{N}}$$

where S = the standard deviation of a sample,
Σ means "sum of,"
X = each value in the data set,
$\overline{X}$ = mean of all values in the data set,
N = number of values in the data set.

Having determined the values of volumes of HCl needed to neutralise the excess of the alkali. The amount of moles of the acid can and should be calculated. (C_{HCl}

Number of moles of hydrochloric acid (n_{HCl})

$= V_{av} \times c_{HCl} = 0.0159\ dm^3 \times 1\ mol\ dm^{-3} = 0.0159$ moles

The reaction occurs due to the following equation:

$$HCl_{(aq)} + NaOH_{(aq)} \rightarrow NaCl_{(aq)} + H_2O_{(l)}$$

The molar ratio of the acid to the base is 1:1, the amount of the base neutralized is equation to the value calculated above. Therefore:

$$n_{\text{excess of NaOH}} = 0.0159 \text{ moles}$$

Polopirin is a monobasic acid, so $n_{aspirin} = (\ n_{base} - n_{\text{excess of NaOH}}\ )$

(n_{base} stands for the number of moles of sodium hydroxide taker initially)

$n_{base} = 0.020\ dm^3 \times 1.0\ mol = 20 \times 10^{-3}$ mole, hence:

$n_{aspirin} = 20 \times 10^{-3}$ mole – 0.0159 = 0.0041moles

Molar mass of Polopirin ($M_{aspirin}$)

$= 9 \times 12.01\ g \cdot mol^{-1} + 8 \times 1.01\ g \cdot mol^{-1} + 4 \times 16.00\ g \cdot mol^{-1} =$

$180.157\ g \cdot mol^{-1}$

Mass of pure aspirin ($m_{aspirin}$) = ($n_{aspirin} \times M_{aspirin}$) =

$0.0041\ mole \times 180.157\ g \cdot mol^{-1} = 0.74\ g$

The uncertainties of the above results should also be calculated:

1) $\Delta n_{excess} = n_{excess} \times (\Delta \frac{V_{av}}{V_{av}}) = 0.00159 \times (0.1\ cm^3 / 15.9\ cm^3) =$ 0.00001 mole

2) $\Delta n_{base} = n_{base} \times (0.1\ cm^3 / 10\ cm^3) = 20 \times 10^{-3} \times (0.1\ cm^3 / 20\ cm^3) =$

$= 0.0001$ mole

3) $\Delta n_{aspirin} = \Delta n_{excess} + \Delta n_{base} = 0.0001 + 0.00001$ mole

$= 0.00011$ moles

Uncertainty of mass calculations:

$\Delta m_{aspirin} = m_{aspirin} \times (\frac{\Delta n_{aspirin}}{n_{aspirin}}) = 0.74 \times (0.00011$ mole $/ 0.0041$ mole)

$= 0.02$ g

There fore the final result can be expressed as:

$m_{aspirin} = (0.74 \pm 0.02\ g)$ g

Now the percentage purity can be obtained:

Purity $= m_{aspirin} / m_{tablet} = 0.74 / 1.442 = 51\%$

This results also has its uncertainty:

$\Delta mv_{av} / m_{tablet} + \Delta m_{aspirin} / m_{aspirin}$) x purity = (0.01/ 1.442 + 0.02 g / 0.74) x 51 % = 3%

Finally the purity of the tablet can be said to be (51 $\pm$ 3 %) of the sample.

Conclusion and Evaluation

The result obtained in this experiment states that in 1.44 g sample of polopirin bought in the chemist's there is about 0.74 g of pure acetylsalicylic acid, which makes up about 51% of the mass of the tablet. The value, including the experimental uncertainty, lies between 48% and 54%. The accepted value for the specified amount was labelled as about 70% pure.

We can see from our calculations and the tables above, that the measurements, and all of them, involve a very low probability of random error, and that is what resulted in low standard deviations. Not only standard deviation but also percentage uncertainty were all on the lower level. That is can by due to precise weighing and measuring devices as well as precision while doing or undertaking the titration process.

During titration labs there is always a chance that one can over-titrate, this means that you continue to add the base even after the required actual colour change has taken place already. This can be due to the eyes of the observer as they are not 100% focused. The person can easily miss the exact moment of neutralisation. Such mistakes introduce slight error to results. However this cannot be neglected. In this practical in it was vital and done that this error was reduced to all part, because of the number of trials done. The more number of trials, is a proper way to minimise the significance of the sources of random error. All of these clearly show us that the result we obtained was a rather precise one.

Table 7: Table for Evaluation

Type of Problems	Improvement
Improving Precision	More and more trials could be done.
Impurities	All the materials should be cleaned before the experiment. It should be rinsed thoroughly before the practical , especially the burette.
Improvement of Method	Because the reaction occurs slowly, it is a good idea to heat the mixture before titrating to let all of the aspirin neutralised. Actually, we did that and that can be qualified as a positive.
Ways to reduce error of Neutralisation Moment	There are many ways in which this can be done they are listed in the paragraph above.
Random Error	Readings from the burette as well as the weightings can be repeated several times and averaged for better results.

As it is impossible to eliminate the human factor in titration process it can be as advised to try to make it easier to find the exact moment in the titration process. This can be done any many simple ways. First way is having more than one person to be looking at it, because attention spans and reaction time one a person can be faster than the other person. Second way, is having a dark piece of cardboard underneath the breaker, which would allow for greater accuracy when looking at the solution at the right angle to its surface. Thirdly, the solution should be stirred

during the titration to allow the based spread evenly. The third solution of stirring, which was actually applied to our experiment and it worked properly.

Apart from that distilled water can be spread on the walls of the beaker from time to time in order to ensure that no drops of base are left outside of the solution. After the acid is thought to be fully neutralised, it is advisable to consult the colour change with other experimenters. Finally, in order to reduce the random errors, the reading from the burette as well as the weights can be repeated several times and average to get proper results and more data. The more data you have the more you can work with it.

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