

Then then ASA in Analysis

What?



The Chemistry of Aspirin

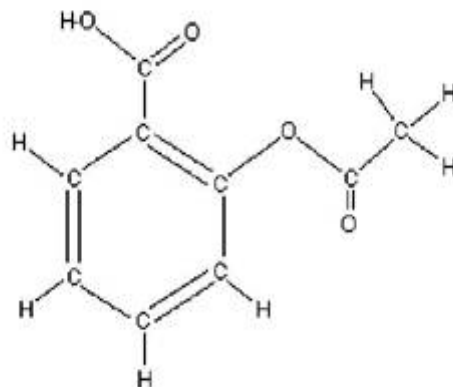
Aspirin, one of the first drugs to come into common usage, is still mostly the widely used in the world - approximately 35,000 metric tonnes are produced and consumed annually, enough to make over 100 billion standard aspirin tablets every year.

Aspirin, also known as 'acetylsalicylic acid', has a chemical formula of $C_9H_8O_4$.

How?

The chemical structure of aspirin:

**Figure 1: Structural Formula of Aspirin
(Acetylsalicylic Acid or ASA)**



Chemical name	2-(acetyloxy)benzoic acid
Chemical formula	C ₉ H ₈ O ₄ C ₆ H ₄ (OCOCH ₃)COOH
Synonyms	2-acetyloxybenzoic acid 2-acetoxybenzoic acid acetylsalicylate acetylsalicylic acid O-acetylsalicylic acid
Molecular mass	180.57 g/mol
CAS number	50-78-2
ATC code	B01AC06
Density	1.40 g/cm ³
Melting point	136°C (277°F)
Boiling point	140°C (284°F)
SMILES	CC(=O)OC1=CC=CC=C1C(=O)O

Its Existence

Aspirin is a painkiller, anti-inflammatory, antipyretic and is an inhibitor of platelet aggregation. It inhibits fatty acid cyclo-oxygenase by acetylation of the active site of enzyme and the pharmacological effects of aspirin are due to the inhibition of the formation of cyclo-oxygenase products including prostglandins, thromboxanes and prostacyclin. Aspirin is prepared by chemical synthesis from salicylic acid, by acetylation with acetic anhydride.

Aspirin Timeline

From origins as an herbal folk medicine - through its extended life as a front line painkiller – and now to its potential applications as a life saving preventive medicine, aspirin's role is constantly changing. All these developments have come from the efforts of thousands of doctors and scientists from across the world

Heart attacks

Aspirin is now accepted as an important weapon in the prevention of heart disease. 20,000 US doctors showed that aspirin reduced the risk of coronary thrombosis by 44 per cent. A single dose of 300 mg is now recommended for patients in the acute stages of a heart attack followed by a daily dose of 75-100 mg. A similar low dose treatment regime

is recommended for patients with angina, a history of heart problems or who have undergone coronary bypass surgery.

Strokes

A trial reported in the Lancet this year is the latest in a sequence of studies showing that aspirin reduces the risk of strokes in patients with 'early warning signs' of transient ischaemic attacks. Further trials showed a small but definite benefit in reducing mortality in those patients (T.I.A.'s) in the acute phase of a stroke.

Pregnancy Complications

Pre-eclampsia and foetal growth retardation, both caused by blockages of the blood vessels of the placenta, are two of the commonest complications of pregnancy - there are 50,000 cases of pre-eclampsia in Britain a year. In a trial involving more than 9000 women in 16 countries, a daily dose of 60 mg aspirin reduced the risk of pre-eclampsia by 13 per cent. Earlier research suggested that the benefits were even greater.

Diabetes -

Blindness, coronary artery disease, stroke and kidney failure are all common complications of diabetes resulting from impaired blood circulation. The benefits of taking one aspirin a day are now so widely accepted that it is considered unethical to perform placebo controlled trials to prove the case.

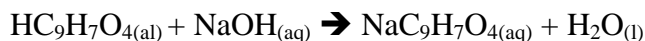
Alzheimer's disease

Some form of dementia affects about one in four people aged 70 years or above. There is some evidence that aspirin may help prevent both the condition resulting from impaired blood flow and the most serious form of dementia, Alzheimer's disease. The latter is believed to be an inflammatory condition similar to arthritis. Aspirin is a highly effective anti-inflammatory drug and a preliminary study found a lower than expected incidence in patients with rheumatoid arthritis, who frequently have to take aspirin over a prolonged period. An important trial in South Wales is following the fortunes of 400 men and studying the factors (including aspirin consumption) that may determine the incidence of dementia. However, volunteers are still being signed up for the trial and conclusive results will not be ready for some time.

Introducing The Importance

The world's most commonly used pain-relieving drug, the Acetylsalicylic acid is generically known as ASA or Aspirin. It is an acetyl derivative of salicylic acid that is a white, crystalline, weakly acidic substance, with melting point 137°C. It is useful in the relief of headache and muscle and joint aches. Aspirin is also effective in reducing fever, inflammation, and swelling and thus has been used for treatment of rheumatoid arthritis, rheumatic fever, and mild infection. $\text{HC}_9\text{H}_7\text{O}_4(\text{s})$ (ASA) is an organic acid (carbon chain) similar to that of acetic acid, $\text{HC}_2\text{H}_3\text{O}_2(\text{l})$ reacts with strong bases such as sodium hydroxide in the same way. There are several complicating factors that arise from the

nature of ASA tablets. For example, all tablet medications contain other “inert” ingredients besides the “medicinal” ones – and some of these other ingredients are not soluble. ASA is a large molecule with only one OH group to provide hydrogen compound as soluble in alcohols, ethers, and chloroform. Some tablets of this type have special coatings or are “buffered,” meaning that other compounds are added that can and do interfere with ASA’s reaction as an acid. The use of a solvent other than water to dissolve the ASA tablet is also extremely complicated. The formula for this chemical reaction is:



Introduction:

The main purpose of this task is to perform an investigation to accurately determine the ASA content of an over-the-counter pain reliever, and to compare the result with the quantity stated on the product label. In doing so we can use the procedure of titration. Titration is a common laboratory technique used to determine the concentration of substances in solution. It is reliable, efficient, and economical technology that is simple to use. A known volume of the sample to be analyzed is usually transferred into an Erlenmeyer flask. The solution in the buret (called the titrant) is added drop by drop to the sample. Alternatively, the standard solution could be in the flask, so the solution of unknown concentration would be the titrant. The titrant is added drop by drop until the reaction is judged to be complete. To help us identify this point, we select an indicator that changes the color when the reaction is complete. The point at which the indicator changes color is called the endpoint. This is at, or close to the point at which the titrant and sample have completely reacted.

Purpose :

To perform an investigation to accurately determine the ASA content of an over-the-counter pain reliever and to compare the result with the quantity stated on the product label.

Hypothesis:

Based on the manufacturer's claim on the label of the bottle of ASA, I predict that the experiment will be 325 mg.

Safety:

- Safety goggles and apron must be worn.
- Acetylsalicylic acid must be stored at room temperature.

- Should ingestion occur with Sodium hydroxide, severe pain burning of the mouth, throat and esophagus. Vomiting, diarrhea, collapse and possible death may result.
- Irritation of the nose, throat and lungs would occur due to the corrosive nature of sodium hydroxide.
- Working stations and area must be cleared.
- Any spills or breakages must be reported to the teacher immediately.

Materials:

- 2 burettes
- 1 dropper
- Sodium hydroxide (30 mL) 0.02M
- 1 Erlenmeyer flask
- 50 mL of water
- 50 mL graduated cylinder
- Phenolphthalein
- 2 labeled beakers
- Retort support stand and retort rod
- 1 ASA tablet

Procedure:

1. Safety goggles and aprons were worn.
2. A tablet of acetylsalicylic acid was obtained and put into a clean beaker labeled "acid."
3. The acetylsalicylic acid tablet was dissolved in 25 mL of water obtained in a graduated cylinder.
4. The acetylsalicylic acid dissolved in 25 mL of water was poured into a clean buret labeled "acid."
5. 5 mL of $\text{NaOH}_{(aq)}$ was obtained in a clean, dry 100 mL labeled beaker.
6. Buret was set up with $\text{NaOH}_{(aq)}$, following the accepted procedure for rinsing and clearing any air bubbles.
7. Another buret was set up with an ASA tablet, as in step 4.
8. 5 mL of $\text{NaOH}_{(aq)}$ was dispensed from the buret into a cleaned Erlenmeyer flask.
9. 1 drop of phenolphthalein indicator was added to the Erlenmeyer flask.
10. Initial buret reading was recorded to the nearest 0.1 mL.
11. $\text{NaOH}_{(aq)}$ was titrated with the sample until a single drop produced a permanent change from faint pink to colourless.
12. Final buret reading was recorded to the nearest 0.1 mL.
13. Steps 5 to 9 were repeated until three consistent results were obtained.
14. Workstations were cleaned and student's hands were washed.

Observations:

	INITIAL	FINAL
Base	27.5	32.5
Acid	31.6	34.4

	INITIAL	FINAL
Base	32.5	37.5
Acid	34.4	37.4

	INITIAL	FINAL
Base	37.5	42.4
Acid	37.4	40.4

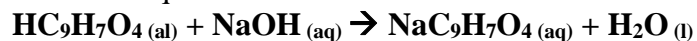
Sources of Error:

- Failure to identify the correct endpoint
- Air bubbles in buret to result in inaccurate readings
- Residue in the equipment could have slowed down or sped up the process
- Inaccurate calculations

Discussion

Mass of ASA = 650 mg

- 1) Write the equation for the neutralization reaction.



- 2) What is the name of the salt formed?

The salt formed is sodium acetylsalicylate.

- 3) Use stoichiometric calculations to determine the mass of ASA in a tablet.

Average volume of acid ($\text{HC}_9\text{H}_7\text{O}_4 (\text{al})$) used

$$\frac{(2.5 + 2.7 + 2.7) \text{ mL}}{3} = 2.63 \text{ mL } \text{HC}_9\text{H}_7\text{O}_4$$

Average volume of base ($\text{NaOH} (\text{aq})$) used

$$\frac{(5.1 + 5.2 + 5.0) \text{ mL}}{3} = 5.1 \text{ mL } \text{NaOH}$$

	$\text{HC}_9\text{H}_7\text{O}_4 (\text{al}) + \text{NaOH} (\text{aq}) \rightarrow \text{NaC}_9\text{H}_7\text{O}_4 (\text{aq}) + \text{H}_2\text{O} (\text{l})$						
Molar ratio	1 mol	:	1 mol	:	1 mol	:	1 mol
Volume	2.63 mL		5.1 mL				
Concentration	C		0.2 M				

Molar Mass

$$\text{mm}_{\text{HC}_9\text{H}_7\text{O}_4} = 8(1.01) + 9(12.01) + 4(16) \\ = 180.17 \text{ g/mol}$$

$$\text{mm}_{\text{NaOH}} = 22.99 + 16 + 1.01 \\ = 40 \text{ g/mol}$$

Concentration

$$C_A V_A = C_B V_B \\ C_A(0.00263 \text{ L}) = \frac{0.2 \text{ mol}}{1 \text{ L}}(0.0051 \text{ L}) \\ C_A(0.00263 \text{ L}) = 0.00102 \text{ mol} \\ C_A = \frac{0.00102 \text{ mol}}{0.00263 \text{ L}} \\ C_A = 0.388 \text{ mol/L}$$

Mass of ASA in a tablet

Mass of ASA = X
Molar Concentration = 0.388 mol/L
Molar Mass = 180.17 g/mol
Volume = 2.63 mL

$$C \text{ (w/v)} = \frac{0.388 \text{ mol}}{1 \text{ L}} \times \frac{180.17 \text{ g}}{1 \text{ mol}} \\ = 69.91 \text{ g/L} \\ m = 0.00263 \text{ L} \times \frac{69.91 \text{ g}}{1 \text{ L}} \\ = 0.165 \text{ g} \rightarrow 165 \text{ mg}$$

Therefore, there is 165 mg of ASA in the tablet.

4) Calculate the percentage difference between the predicted and experimented mass.

$$\frac{\text{Actual mass}}{\text{Manufacturer's mass}} \times 100 = \% \text{ difference}$$

$$\frac{165 \text{ mg}}{650 \text{ mg}} \times 100 = 25.38 \%$$

5) Evaluate the prediction and the manufacturer's claim.

The manufacturer's claim was that there was 650 mg of ASA in one tablet. However, when we used titration to test for ASA, we found out that there was only 165 mg of ASA in the tablet. If our results are correct, we have discovered that there really isn't as much ASA in a tablet, and that the manufacturer's claim

is false. However, our results may not be accurate because some of the ASA might have been left on the mortar, pestle, beaker, or even the burette.

Conclusion

We learned how to titrate properly and in the most professional way. While doing this lab report a few unscheduled mishaps occurred. First was, the burette we took was not working properly, and thus we had to use another one. The second mishap occurred three-fourths way into the experiment when our burette containing the aspirin dissolved in acid completely broke. This occurred because the acid ate away at the plastic. We had to quickly do the experiment over again to receive useable results. The second time we redid our titration our measurements were more accurate. We obtained an average of 2.63 mL of volume for the acid. When our calculations were complete we discovered that there was only 165mg of ASA in one tablet. However the manufacture claimed that there was 650mg of ASA in one tablet. This would make the percentage difference of the predicted and the experimented mass 25.38%. This is very off from what the expected mass was, as stated in the hypothesis. Perhaps there were errors in the accuracy of the experimental design. Some of the ASA could have been left in the mortar or on the pestle, beaker, or even the buret. Or perhaps this is a mistake made in the calculations, or in the manufacture's claim. We however know that the laboratory experiment was done to the best of our abilities. All in all, this was a great experience, a good lab and a great way to understand the nature of acids and bases through hands on work.