

Volumetric Analysis: Back Titration of Aspirin

Assessment Design Criteria

IAE2	Obtains records, and represents data, using appropriate conventions and formats.	KA1	Demonstration of knowledge of range of chemistry concepts
IAE3	Analyses and interprets data and evidence to formulate logical conclusions with detailed justification.	KA4	Communicates knowledge and understanding of chemistry, using appropriate terms, conventions and representations
IAE4	Evaluates procedures and discusses their effect on data.		

Task description

Students carry out an investigation to find the dosage of acetylsalicylic acid in an aspirin tablet. Students will write a scientific style report which includes their findings, their calculations to determine the amount of aspirin in moles, grams and %mass per tablet. They will also answer several given questions as part of their analysis and evaluation.

Conditions

Students will carry out the task under teacher supervision. Students will prepare a written report to be submitted a week after the practical.

Preamble

At the turn of the 20th century the discovery and testing of medicines was a largely unregulated process. Many new compounds were given to patients almost immediately after synthesis or discovery. In contrast, the development of modern pharmaceuticals is a long and expensive process typically taking over a decade. Chemistry is used at all stages to develop the synthesis of the pharmaceutical and determine its purity.

Before being administered to potential patients, chemists need to know how the pharmaceutical works, how safe it is, and what dosage is required. How the pharmaceutical works is initially investigated using an isolated enzyme or cellular systems (cell cultures) before being trialled in animal models and ultimately in volunteer patients. Clinical trials are used in the later stages to see if a new pharmaceutical works in one set of patients compared to the effects of a placebo on another group.

Aspirin is a commonly available, regulated analgesic. This task will involve using analytical chemistry techniques to determine the amount of acetylsalicylic acid in an aspirin tablet.

Report Structure and Guidelines

Introduction*

- State clearly the purpose of the investigation.
- Include relevant scientific concepts - including an explanation of the purpose of back titrations and why it is used in this context.
- A description of factors that were controlled during the experiment.
- Identification of pertinent factors that could **not** be controlled and explain why not.

Materials/Apparatus

- A list of materials used.
- Clearly labelled photographs of the experimental apparatus should be included.

Procedure

- A step-by-step procedure.
 - As part of this you can justify why certain steps are performed and how that affects the results.

Safety Considerations

- A table that identifies safety risks or ethical considerations and **how** they were managed.

Results

- Record titration results in an appropriate table.
- Include relevant observations.

Calculations:

- Calculations should be neatly laid out with the steps clearly explained.
- As the steps will need to be carried out for each of your runs, you may show how you calculated your answers for one titration and then just give the results for the others.
- You will need to:
 - Write the balanced equations for the relevant reactions.
 - Determine the moles of base added to each flask.
 - Determine the moles of base reacted with the acid.
 - Determine the moles of base in excess.
 - Determine the moles of aspirin in each sample.
- You need to determine the moles, mass (grams) and %mass of acetylsalicylic acid per tablet.

Analysis and Evaluation*

Your discussion should include:

- Critical and systematic analysis of the data in the context of the scientific concepts being investigated.
- Discussion of the precision and accuracy of the results.
- Discussion that addresses the following prompts:
 - Ethanol is slightly acidic and will react with NaOH. Justify how would this affect your results.
 - Suggest why a burette was used to add the excess NaOH instead of a measuring cylinder.
 - The reaction of aspirin with NaOH has two steps, a fast deprotonation step involving one hydroxide molecule and a slow hydrolysis step involving another hydroxide molecule.
 - What factor(s) in this experiment are used to try to ensure the hydrolysis goes to completion?
 - How would your results be affected if the hydrolysis was incomplete?
 - Briefly outline how we could run an experiment to see if hydrolysis was complete.

Conclusion*

- Formulate a logical and justifiable conclusion that relates to the purpose of your practical investigation.
- Identify any limitations to your conclusion.

***included in word count**

Aspirin Titration Laboratory Procedure.

Materials:

- Aspirin tablets (Astrix brand 100mg)
- 0.1M NaOH (*standardised*)
- 0.1M HCl (*standardised*)
- Phenolphthalein indicator
- Ethanol
- 250mL Erlenmeyer flasks * 3+
- Glass Funnel
- 70°C Water Bath (70° Max)
- Weight boats and spatulas.
- Electronic Mass Balance
- 50ml Burette
- 20ml Measuring Cylinder
- Retort Stand & Burette Clamp

Procedure:

Note: Make sure to have labels, pen and paper to record data as you go along.

Sample Preparation

1. Place 7 tablets in a weigh boat. Record the total weight and then the average weight per tablet.
2. Crush the tablets to a fine powder in the weigh boat with the back of a spatula.
3. Into clean weigh boats, weight out at least three separate lots of at least 0.2 grams of crushed tablet (record the actual weight of each sample to 3 decimal places).
4. Prepare 20ml of ethanol for each sample.
5. Empty the weigh boat into a 250 mL Erlenmeyer flask and use some of the 20ml to wash the last bit of sample in the weigh boat into the flask and then add the remaining ethanol to the flask so that each sample has 20mL.
6. Gently swirl the flask to dissolve the tablet powder in the Ethanol

NB: Labelling your flasks and recording specific details for each is essential- a table is strongly recommended.

Determination of Excess NaOH volume.

1. Add 3 drops of phenolphthalein indicator to each sample.
2. Titrate one sample (preferably the one with the most aspirin) using the NaOH solution until it reaches the first permanent cloudy pink colour and record the titre value (if you overshoot a little it's okay, just record the volume)
 - a. The quicker acid – base reaction consumes one mole of hydroxide per mole of aspirin.
 - b. The slow hydrolysis reaction also consumes one mole of hydroxide per mole of aspirin.
 - c. For the complete titration we will need twice the amount of NaOH you have already used plus excess to back titrate. Calculate as follows:
(NaOH used x 2) + 5mL.
3. Using your burette, continue to carefully add NaOH up to the required total, then also accurately add this same volume to the other two samples.
 - a. Refill your burette as required.

Speeding up Hydrolysis

1. Cover the neck of each sample flask with gladwrap to reduce evaporation, and heat each in a 70°C water bath for 20 minutes and then allow to cool.
2. If the samples are colourless, wash sides of the flask with a little distilled water and add a few more drops of phenolphthalein and swirl. If it remains colourless, carefully add 10mL more of NaOH to each sample and reheat.
 - a. Make sure to add this 10mL of NaOH to your record of your total.

Back Titration with Acid

1. Wash the burette and fill with HCl (*or use a second clean burette*)
2. Carefully titrate each sample until the solution just turns from pink to clear and stays a slightly cloudy white.
3. If the Aspirin sample has been fully neutralised and hydrolysed, then the titre should be close to the excess amount of NaOH that was added (approx. 5ml) so **be careful and titrate drop-wise so as to not over-shoot the end point.**
4. Record your titres.
 - a. Remember- you will not get concordant values due to having potentially different sample weights and NaOH additions. However, the calculated results should be close to each other if you have been careful.