

Comparison of the active ingredient and purity of two different commercial Aspirin tablets – Session 2

Summary of Session 1

During Session 1, you discussed methods for carrying out a rapid and simple extraction of the active component in Aspirin, carried out an agreed method for the extraction, and evaluated the outcomes. The yield of the extracted material was determined, but this did not reveal whether a truly *quantitative* extraction was achieved. Some elementary chemical tests revealed the presence of some salicylic acid, but the presumed main component, acetyl salicylate, was not explicitly tested for. The *selectivity* of the extraction was also not determined.

Session 2

The aim of this session is to use an extraction method which should be more quantitative, together with a simple titrimetric method for determining the amount of active ingredient in the extract.

Working with the same partner as Session 1, discuss your findings for the extraction and analytical methods that you have researched as the preparation for this session (Session 2 pre-laboratory exercise).

You should consider the following in your discussions:

- The experimental procedure should allow you to obtain a higher yield than the simplified method used in the previous session.
- The experimental procedure should take no longer than two hours.
- You may only use the glassware and solvents available to you.
- Your titrimetric method should take into account the chemical structure of acetyl salicylate (How might the method exploit the specific functional groups?)
- As with Session 1, you will need to decide how to best record your results in order for you to be able to evaluate the session aims.

You will have **15 minutes** to discuss your experimental procedures before discussing your conclusions with a member of staff. You will then be in a position to carry out an agreed extraction procedure and subsequent analysis.

Method to carry out a Soxhlet extraction of acetyl salicylate from Aspirin

The class will be divided into two. As a pair, you will carry out the extraction and analysis on **one** sample of Aspirin only.

Soxhlet extraction

You will be performing a Soxhlet extraction on your sample. Accurately weigh 7 Aspirin tablets to 4 decimal places, and crush the tablets on a piece of hard filter paper using a spatula. Place the powder in the Soxhlet thimble ready for extraction. After recording the weight of the round bottomed flask, add 150 cm³ of ethanol. Assemble the apparatus and turn on the heating mantle. Once the ethanol is boiling gently, continue the extraction for 30 mins (about 4-5 cycles) before turning off the heater and allowing the solution to cool. When at room temperature, remove the round bottomed flask and evaporate the ethanol using a rotary evaporator.

Re-weigh the flask and calculate the yield based on the mass of extract. Compare this yield to that obtained from Session 1 and to the amount you would have expected from the packaging (you may also want to express your yield as a percentage).

In order to analyse your extract further, you will need to divide it into two halves.

Titration of extract with NaOH

Transfer approximately half of your extract into a vial, ensuring that you obtain an accurate mass. Add 20 cm³ of distilled water to the remaining extract in the round bottomed flask. If the extract does not completely dissolve, add sufficient ethanol (approx. 20 cm³) to ensure that it all dissolves. Add 3 drops of phenolphthalein indicator and titrate the resulting solution with 0.1 M sodium hydroxide. The end point is observed when a faint pink colour persists for at least 30 seconds after swirling.

Record the initial and final volumes of NaOH in the burette and hence the volume used in the titration.

Chemical tests

Carry out chemical tests (a) and (b) on the following and make notes of all your observations:

- A small sample of extract
- The used Soxhlet thimble
- You may also find it useful to refer back to your results from the previous session (including results for a tablet which has not been extracted, and authentic salicylic acid)

Note: Quantities of compounds / reagents described for these qualitative tests are guidelines only.

(a) Ferric chloride test for a free phenolic group (-OH):

The addition of ferric chloride to salicylic acid produces a specific colour following a reaction with aqueous ferric $[\text{Fe}(\text{H}_2\text{O})_6]^{3+}$ ions. The oxygen atoms of the carboxylic acid group ($-\text{CO}_2\text{H}$) and the phenol group ($-\text{OH}$) concomitantly form a complex with $[\text{Fe}(\text{H}_2\text{O})_6]^{3+}$. This complex has an intense violet colour. With acetyl salicylate, the ($-\text{OH}$) group of salicylic acid has been replaced by a ($-\text{O}-\text{COCH}_3$) group, which prevents the formation of the violet-coloured complex.

Add 3 drops of ferric chloride solution to 0.2 g of a crushed Aspirin tablet in about 2 cm³ of water. If you observe a violet colouration, this indicates the presence of the free phenol group ($-\text{OH}$). Repeat the test using 0.1 g of salicylic acid and 0.1 g of your extracted samples. Note any differences between the intensities of the violet colour and consider the significance of these differences

(b) Test for starch

Add 1-2 drops of Iodine-KI reagent to a small amount of your sample. Samples containing starch will produce a blue-black colour. If starch is not present, the colour will remain orange/yellow. What would the presence of starch in your extract indicate about the selectivity of the extraction? What impact might this have on the % extraction calculations?

Session 2 Post-laboratory exercise

Summarise your findings and make comparisons with the results and outcomes of Session 1. In order to interpret your titration data, you will need to write down the equation for the reaction of acetyl salicylate with NaOH. Include the following data and calculations in your summary:

From gravimetric data:

- Yield of extract (mg)
- % yield of extract (comparison of yield with packaging information)

From titrimetric data:

- Titration data and mass of acetyl salicylate in extract
- % of acetyl salicylate in the extract
- Mass of acetyl salicylate in the original sample and an average mass per tablet
- A comparison of the mass of extracted acetyl salicylate with the dose indicated by the packaging

Having assessed your data from Sessions 1 and 2, you should now be in a position to evaluate the quantitative nature of both extraction methods and have a more specific identification and quantification of the active ingredient from the titration data.

Further discussion points (in-class):

Firstly, you may have found that your extracts gave a positive test for salicylic acid even though the ferric chloride test yielded a less intense colouration than when the test was carried out on pure salicylic acid. Secondly, the outcome of the titration analysis may need to be reconsidered in light of the chemical structures of acetyl salicylate and salicylic acid.

Refine your evaluation of the outcomes from Session 2 by discussing these two further points. Decide upon the outcomes which might be achievable in the next session.

Session 3 pre-laboratory exercise

In Session 3, you will carry out a more rigorous and explicit identification of your extract obtained in Session 2. You will be using analytical techniques which you should already be familiar with including:

- Melting point measurement
- Infra-red spectroscopy (IR)
- Functional group identification via chemical tests
- Gas chromatography (GC)

In order to interpret the data that you will collect during Session 3, you will need to find *literature data* for both acetyl salicylate and salicylic acid and address some further points as described below:

1. Find the melting points of acetyl salicylate and salicylic acid. Why might these data be useful within the context of the current investigation?
2. Identify the functional groups present in acetyl salicylate and salicylic acid, and predict the positions and strengths of the absorption bands that you would expect for these groups using Infra-Red spectroscopy. What limitations might this method have within the context of the current investigation?
3. Describe why it might be useful to investigate your extract using gas chromatography (GC). How would you use the results of the GC analysis to provide more detailed information about the composition of your extract?
4. What is the purpose of carrying out functional group tests on your extract?

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