

ASPIRIN STRENGTH

Determining the aspirin content of tablets



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Languages available

English, Hungarian, Slovenian

Summary

You need to know how to identify the most appropriate analytical method for tackling a particular analytical problem. There are many types of analytical methods and you need to know their limitations. Imagine that you have to choose between two analytical methods available in your lab, to measure the active ingredient of aspirin (acetylsalicylic acid).

Activity type A

Use of scientific knowledge and understanding to solve problems

Working in teams to solve problems

Techniques

Titration

UV-VIS spectrophotometry

Field

Pharmaceuticals

Time

Practical lessons: 180 minutes

Theory lessons: 45 minutes

Out of class time: 45 minutes

StandardBase procedures

Determination of aspirin using acid/base titration

StandardBase techniques

Volumetric and gravimetric analysis

Ultraviolet and Visible (UV-VIS) Absorption Spectroscopy

Other resources

Gedeon Richter plc , Budapest, Gyömrői u. 19-21, Hungary; www.richter.hu

<http://en.wikipedia.org/wiki/Aspirin>

www.chemsoc.org/PDF/LearnNet/rsc/Aspirin_full.pdf

<http://services.juniata.edu/ScienceInMotion/chem/labs/ap/aspirin.doc>

Student's document

ASPIRIN STRENGTH: Determining the aspirin content of tablets

Analysts are asked to identify what substances are present in samples and how much there is. The samples may have many different origins (environmental samples, drugs being synthesised in the laboratory etc.). You need to know how to identify the most appropriate analytical method for tackling a particular analytical problem. There are many types of analytical methods and you have to know the limitations of your methods.

Commercial aspirin tablets are not 100% acetylsalicylic acid, they contain binder as well. Moreover, moisture can hydrolyse aspirin, so you may be able to detect a vinegar odour in aspirin if it has been exposed to moisture for a period of time. Imagine that you have to choose between two analytical methods available in your lab, to measure the active ingredient of aspirin (acetylsalicylic acid).

Your brief

You are the leader in a laboratory that carries out routine analyses on pharmaceutical products. You have to choose titration or the spectrophotometric method available in your lab, to measure the active ingredient of aspirin (acetylsalicylic acid). Which method is more accurate (and/or simple)?

Your investigation

Consider how you can determine acetylsalicylic acid.

There are several possibilities but in your laboratory only:

1. Acid/base titration
2. UV-VIS spectrophotometry.

At least two groups will be needed to carry out this activity (see **Student sheet: Procedures and data**).

Group A will measure the active ingredient of aspirin using acid/base titration.

For full details, see www.standardbase.com

Group B will measure the active ingredient of aspirin using spectrophotometry.

For full details, see

<http://services.juniata.edu/ScienceInMotion/chem/labs/ap/aspirin.doc>

Alternatively, your teacher will supply the necessary procedure.

Estimated time

- Gathering information from library and Internet - 45 minutes
(A good starting point is www.standardbase.com)
- Laboratory work to determine the content of acetylsalicylic acid from tablets (or 'home-made aspirin') using a chosen analytical method - 4 x 45 minutes
- After the practical work, evaluating and comparing results and methods for the two different analytical methods - 45 minutes

A risk assessment must be carried out before starting practical work.

Your findings

Compare your result with that given by the manufacturer [on the packaging you will find e.g. "Each tablet contains 300 mg aspirin; also contains microcrystalline cellulose (E460), hydroxypropyl methyl cellulose (E464)"].

- Do your results match with the analysis of the manufacturer?

- Calculate the mean and standard deviation of your analysis.
 - How confident are you on the reliability of your findings?
 - What are the possible errors in your results?
 - Compare your result with other groups' results. Make a comparison table.
- Do not forget to provide a summary of the results for your teacher.

Write a report (1-2 pages) about your investigation.

- Make a table to compare the two analytical methods. You might include: analytical method, main steps of the procedure, amounts of chemicals used, equipment, active ingredient of aspirin as a percentage giving mean and standard deviation

Student self assessment

Complete the checklist below. If there are more than three "NO" answers, please consult your teacher.

Using scientific knowledge and understanding to solve problems

1. Can you search information after putting in a keyword on the Internet?
2. Have you found useful information other than the suggested articles and websites?
3. Can you summarise the advantages and disadvantages of your chosen analytical method?
4. Do you know the principle of your chosen method?
5. Do you use a spectrophotometer in practice?
6. Did you understand the meaning of accuracy and standard deviation?

Working in teams to solve problems

1. Did you share the work between the members of your group?
2. Did you help other team members with their problems?
3. Can you evaluate the reliability of your result?
4. Did you compare your result with the expected (e.g. acetylsalicylic acid content on the box) and with other groups' results?

In 50-100 words, describe how you worked as a team and give examples of how you supported others in your group or were supported by them. What is the advantage of working in a team?

Student sheet: Procedures and data

Tasks

- Gather information
- Discuss the information and suggested measuring method with your teacher
- Discuss with your teacher the required/available chemicals, instruments, glassware
- Prepare stock – standard – solutions/titrant
- Prepare calibration curve/carry out titrations
- Prepare the sample for measuring/titration
- Measure samples
- Collate final result(s), report, comparison table

Task 1

Acid/base titration

Use the procedure *Determination of aspirin using acid/base titration* from www.standardbase.com

Carry out at least 3-5 measurements.

Task 2

Spectrophotometric method

Your teacher will supply you with the detailed procedure for the UV-VIS spectrophotometric analytical method, but first look at the procedure on the Internet.

Carry out at least 3-5 measurements. Remember to neutralise the excess NaOH after the hydrolysis of aspirin by adding 0.1 mol/dm³ HCl solution until the pH = 5; otherwise you will not be able to analyse it by spectrophotometry because NaOH reacts with FeCl₃ and forms Fe(OH)₃ precipitate.

If necessary, dilute the sample to fit into the calibration curve.

Some data on SA (Salicylic Acid) and ASA (Acetylsalicylic Acid) relevant to this activity:

Compound	Salicylic Acid SA	Acetylsalicylic Acid ASA
Formula	C ₇ H ₆ O ₃	C ₉ H ₈ O ₄
Molar mass (g/mol)	138.12	180.15
K _a	1.08 × 10 ⁻³	2.72 × 10 ⁻⁵
pK _a	2.99	4.57
Solubility in water (g/100 cm ³)	0.18	0.25

Teacher's document

Overview

In this activity, students will:

- use scientific knowledge (titration, Lambert-Beer's law, calibration curve)
- use acid/base titration and spectrophotometric methods
- compare analytical methods
- use the Internet, gathering information
- work in teams to solve problems.

Developing student's skills

Students learn to:

- use knowledge and understanding to tackle scientific problems
- observe, measure, analyse and evaluate scientific data
- work with others and work in teams to solve problems.

Organising and managing the class

Minimum group size: 2

Number of groups: 2

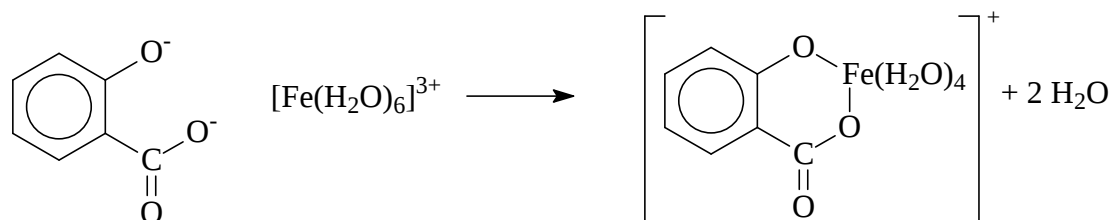
Session plan for teachers

- Teacher's introduction to the scenario (10 minutes)
 - Outline the available equipment (and chemicals)
 - Statement of expected output (content, format)
- Collecting information, from library and Internet, and diagnostic assessment with questions related to the subject (35 minutes)
- Practical work preparing the aspirin sample and measuring the purity using two different analytical methods (4 x 45 minutes)
- Making final report, comparing the results and methods (45 minutes)

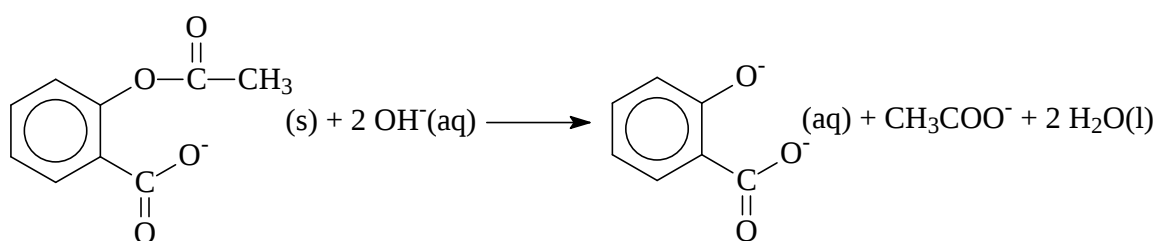
Technical notes

(These relate only to the spectrophotometry procedure)

The method is based on the observation that salicylic acid forms a purple (violet) coloured complex with Fe(III) ions.



The acetyl group in aspirin (ASA) prevents formation of a similar complex, so you have to decide whether you wish to determine only the very small amount of salicylic acid in aspirin or you wish to determine the salicylic acid (SA) after hydrolysis of acetylsalicylic acid (ASA).



▪ Equipment and materials

Materials and their CAS numbers

salicylic acid	69-72-7
sodium hydroxide, NaOH	7647-01-0
hydrochloric acid, HCl	1310-73-8
iron(III) chloride	10025-77-1

Instruments

- UV-VIS spectrophotometer
- calibrated analytical balance

Glassware

- 1000 cm³ volumetric flasks (calibrated as Class 'A')
- 250 cm³ volumetric flasks (calibrated as Class 'A')
- 100 cm³ volumetric flasks (not calibrated)
- funnels
- 25 cm³ burette
- 20 cm³ pipette (calibrated as 'Class A')
- conical flasks ("Erlenmeyer flask")

▪ Procedure and solutions

Task 1

acid/base titration

The detailed description for *Determination of aspirin using acid/base titration* is available on [www. standardbase.com](http://www.standardbase.com)

Task 2

VIS spectrophotometric method

Stock solution

Dissolve 250 mg salicylic acid (SA) in 15-20 ml ethanol. After dissolving, add 150 ml distilled water and dilute to 250 ml with distilled water. The stock solution will contain 1 mg/cm³ salicylic acid.

Reagent solution

Dissolve 10 g FeCl₃.6 H₂O in 0.1 mol/dm³ HCl solution, dilute to 100 cm³ with 0.1 mol/dm³ HCl solution.

Standard solutions

Standard solutions for calibration curve should contain 0–100 mg/dm³ salicylic acid. Use 100 cm³ volumetric flasks. Do not make up to the mark yet.

Sample preparation

- After aspirin hydrolysis (see *Task 1*), transfer the correct (tablet) amount into a 250 cm³ volumetric flask. Do not make up to the mark yet.
- Add 0.1 mol/dm³ HCl solution until the pH = 5.
- To all the volumetric flasks (standards + sample) add 10 ml Fe³ + reagent, then make up to the mark with distilled water (mix well).

▪ Measurement

Measure the absorbance of standard solutions and the sample with the spectrophotometer using a wavelength of 530 nm. The absorbances of the standard solutions are plotted against the SA concentrations in mg/dm³. Microsoft Excel could be used. Calculate the content of ASA (purity of aspirin).

TESTING PRIOR KNOWLEDGE

Choose the correct answer.

1. What is the systematic name of aspirin?
 - a. salicylic acid
 - b. 2-acetoxybenzoic acid
 - c. 2-hydroxybenzoic acid
 - d. N-(4-hydroxyphenyl)ethanamid
2. Has the crude Aspirin product lower melting point than pure aspirin?
 - a. no
 - b. 1-2 °C higher
 - c. same
 - d. yes, significantly
3. What is the best solvent for aspirin?
 - a. water
 - b. acetone
 - c. ethanol
 - d. NaCl solution
4. Which method is the most accurate for quantitative determination of acetylsalicylic acid content?
 - a. measuring melting point
 - b. spectrophotometric
 - c. titration
 - d. thin layer chromatography

Note: answers are given in red. These need to be removed before test is used.

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