

## 73 SERIES SPECTROPHOTOMETER

Application note: A09-009A

### The quantitative determination of the aspirin content of tablets using UV and Visible wavelength spectroscopy

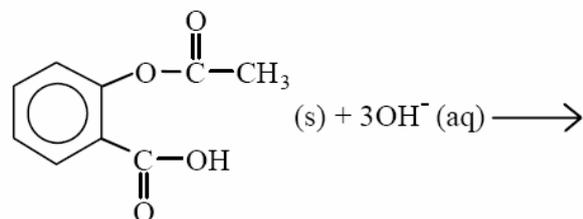
#### ■ Introduction

Since its market introduction in 1899 under the trademark Aspirin®, acetylsalicylic acid has attained a leading worldwide position in the nonprescription treatment of pain, inflammation, and fever. It has proven effective as a general pain reliever, and it is routinely used in a wide range of painful conditions, including head, body and muscle aches, arthritis and many other common ailments.

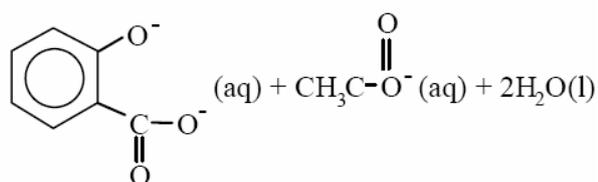
Today, aspirin is also taken by millions of people who benefit from its antithrombotic effect. Patients with a history of cardiovascular disease or stroke benefit from the life saving blood thinning properties of acetylsalicylic acid. The medical community has increasingly recommended routine therapy with low-dose acetylsalicylic acid, which significantly reduces the risk of death from a cardiovascular event (1).

The aspirin content of commercial tablets is routinely determined using the acid/base titration method described in the British Pharmacopoeia (2) but a large number of alternative instrumental methods are also described in literature. This application note will compare two such spectrophotometric methods.

Acetylsalicylic acid is readily hydrolysed in basic medium to yield the salicylate dianion:

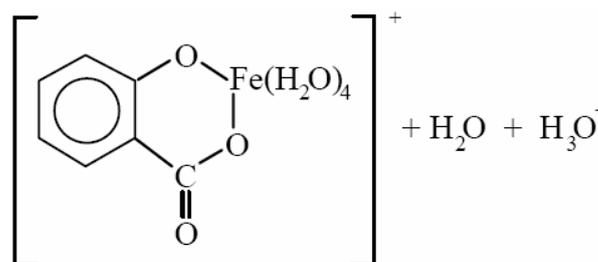
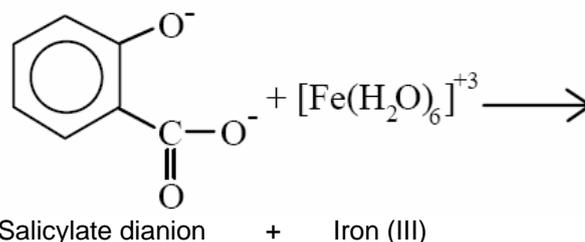


Aspirin (Acetylsalicylic acid)



Salicylate dianion

After acidification the resulting salicylic acid can be determined directly from its absorption maxima at 302nm (Method 1) or it can be derivatised with Fe(III) to produce a violet coloured complex that can be quantified at its absorption maxima of 530nm (Method 2).



Tetraaquaosalicylatoiron (III) complex.

#### ■ Methods

##### Method 1 (3)

##### Reagents

Hydrochloric acid 0.2M  
Sodium Hydroxide 0.2M  
Methanol  
Salicylic Acid  
Citric acid

##### Standard Preparation:-

A stock standard equivalent to 25mg/l aspirin was prepared by dissolving 19.2mg of salicylic acid and 0.1g of citric acid in a minimum amount of purified water. This solution was diluted to 100.0ml with methanol. Working standards were prepared by pipetting 5, 10 and 20 ml aliquots of the stock standard solution into separate 100.0ml volumetric flasks, adding 25ml of 0.2M NaOH and diluting to volume with 0.2M HCl.

##### Sample Preparation:-

Three samples were prepared by weighing a single commercial aspirin tablet (Tesco own

brand), crushing it in a pestle and mortar and then weighing an amount, equivalent to 25mg of aspirin, into a 50.0ml volumetric flask. 30ml of methanol was added and after shaking for 2 minutes the sample was diluted to volume with methanol. 5ml aliquots of each sample were pipetted into separate 100.0ml volumetric flasks and 25ml of 0.2M NaOH was added to each. After standing for 30 minutes each flask was diluted to volume with 0.2M HCl.

## Method 2 (4)

### Reagents

Hydrochloric Acid conc.

Sodium Hydroxide 1M

Salicylic Acid

Potassium Chloride

0.025M FeCl<sub>3</sub>

*Weigh 6.8g FeCl<sub>3</sub>•6H<sub>2</sub>O into a 1.0L volumetric flask and dissolve in 100ml of purified water. Add 3.0ml of concentrated HCl and 12.0g of KCl. Dissolve and dilute to volume with purified water.*

### Standard Preparation:-

A stock standard solution equivalent to 800mg/l aspirin was prepared by dissolving 61.0mg of salicylic acid in 2ml of 1.0M NaOH in a 100.0ml volumetric flask. This solution was then diluted to volume with purified water. Working standards were prepared by pipetting 10, 8, 6, 4 and 2 ml aliquots of the stock standard solution into separate 100.0ml volumetric flasks and diluting to volume with 0.025M FeCl<sub>3</sub>.

### Sample Preparation:-

Three samples were prepared by weighing a single commercial aspirin tablet (Tesco own brand), crushing it in a pestle and mortar and then quantitatively transferring it to a 250.0ml volumetric flask. 10ml of 1.0M NaOH was added and the solutions were heated to boiling. After cooling to room temperature the samples were diluted to volume with purified water. 5ml aliquots of each sample were pipetted into separate 100.0ml volumetric flasks and each flask was diluted to volume with 0.025M FeCl<sub>3</sub>.

### Sample Measurement:-

Aliquots of the samples prepared in both of the described methods were placed into disposable plastic cuvettes (part code 060 230) and analysed using a 7305 spectrophotometer. The Photometrics mode was accessed by selecting the Photometrics icon from the main menu screen and measurements were performed according to the procedure described in the instruments operating manual. Microsoft Excel

was used to tabulate the measurement data and perform the linear regression analysis.

## Results

### Method 1

The absorbance values of the three working standard solutions were measured, the measured values are shown in table 1.

Aspirin equivalent conc. (mg/ml)	Absorbance
0.0125	0.232
0.0250	0.466
0.0501	0.942

**Table 1. Method 1 Standard absorbance values.**

A linear regression of the standard absorbance data gave equation 1.

$$(1) y = 18.895x - 0.0051$$

Re-arranging equation 1 allows the aspirin content of the sample solution to be determined according to equation 2.

$$(2) \text{Aspirin in sample (mg/ml)} = \frac{(\text{Abs} + 0.0051)}{18.895}$$

The aspirin content of each tablet is then determined using equation 3.

$$(3) \text{mg/tablet} = \frac{(\text{aspirin in sample (mg/ml)} \times \text{tablet wt (g)}) \times 1000}{\text{sample wt (g)}}$$

The aspirin content of the three tablets analysed are given in table 2.

Sample	Tablet wt (g)	Sample wt (g)	Sample Abs	Aspirin Content of Tablet (mg)
1	0.3498	0.0291	0.470	302.2
2	0.3422	0.0288	0.469	298.1
3	0.3457	0.0292	0.475	300.8
Average				<b>300.4</b>
Std Dev				<b>2.09</b>

**Table 2. The calculated aspirin content of Tesco own brand tablets using Method 1.**

Tesco own brand aspirin tablets are certified to contain 300mg of aspirin in each tablet. Analysis of three tablets using method 1 gave an average content of 300.4mg with a standard deviation of 2.09. Each tablet that was analysed contained 95.0% to 105.0% of the certified amount, as required by the British Pharmacopoeia (2)

## Method 2

The absorbance values of the three working standard solutions were measured, the measured values are shown in table 3.

Aspirin equivalent conc. (mg/ml)	Absorbance
0.0159	0.139
0.0318	0.273
0.0477	0.410
0.0637	0.545
0.0796	0.684

**Table 3. Method 2 Standard absorbance values.**

A linear regression of the standard absorbance data gave equation 4.

$$(4) y = 8.55x + 0.002$$

Re-arranging equation 1 allows the aspirin content of the sample solution to be determined according to equation 5.

$$(5) \text{ Aspirin in sample (mg/ml)} = \frac{(\text{Abs} - 0.002)}{8.55}$$

The aspirin content of each tablet is then determined using equation 6.

$$(6) \text{ mg/tablet} = \text{aspirin in sample (mg/ml)} \times 20 \times 250$$

The aspirin content of the three tablets analysed are given in table 4.

Sample	Tablet wt (g)	Sample Abs	Sample (mg/ml)	Aspirin Content of Tablet (mg)
1	0.3469	0.516	0.0601	300.6
2	0.3440	0.507	0.0591	295.3
3	0.3400	0.499	0.0581	290.6
Average				<b>295.5</b>
Std Dev				<b>4.97</b>

**Table 4. The calculated aspirin content of Tesco own brand tablets using Method 2.**

Tesco own brand aspirin tablets are certified to contain 300mg of aspirin in each tablet. Analysis of three tablets using method 2 gave an average content of 295.5mg with a standard deviation of 4.97. Each tablet that was analysed contained 95.0% to 105.0% of the certified amount, as required by the British Pharmacopoeia (2).

## Conclusions

The batch of aspirin tablets used in this application note contained 299.5mg of aspirin per tablet when it was tested by the manufacturer (5). The two analysis methods in this application note gave assay results that were in agreement with this value, although method 2 gave a broader range of results (10mg/tablet) than method 1 (4.1mg/tablet).

Further work would be needed to determine if the differences in the results were due to the respective analytical methodologies used in each method or normal random variations in the aspirin content of the individual tablets.

## References

1. Duke University Medical Center (2006, November 17). Low-dose Aspirin Reduces Cardiovascular Events, Analysis Shows. *ScienceDaily*. Retrieved July 21, 2010, from <http://www.sciencedaily.com/releases/2006/11/061116100758.htm>
2. British Pharmacopoeia 2009, Volume III, pg 2305. Formulated Preparations: Specific Monographs, Aspirin Tablets.
3. Adapted from: Sanyal & Dutto, Journal of AOAC International, Vol. 79(6), (1996) pp 1303-05
4. Adapted from: [http://cas.bellarmine.edu/chem116a/lab/aspirin\\_analysis.htm](http://cas.bellarmine.edu/chem116a/lab/aspirin_analysis.htm)
5. Private communication with tablet manufacturer.