



Development of difference spectroscopic method for the estimation of Aspirin in formulation using hydrotrophy

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ABSTRACT

A simple, precise and accurate difference spectroscopic method has been developed for the estimation of aspirin in pharmaceutical dosage form. The proposed method is based on the principle that aspirin can exhibit two different chemical forms which differ in absorption spectra in basic and acidic medium. Since the drug was not freely soluble in the distilled water, 1M sodium salicylate has been used as hydrotropic solubilising agent for aspirin to carryout its spectrophotometric analysis and make the process cost effective. The stock solution was prepared by taking water as solvent. Further dilutions were made by using 0.1M sodium hydroxide and 0.1M hydrochloric acid separately. The maxima and minima in the difference spectra of aspirin were 312nm and 285nm, respectively. Difference in absorbance between maxima and minima was calculated to find out the amplitude. The amplitude was plotted against concentration. Beer's-Lambert's law is valid in the concentration range 0.1-1µg/ml. The result of analysis was validated statistically and by recovery study.

Keywords: Aspirin; Difference spectroscopy; Estimation; Validation.

1. INTRODUCTION

Aspirin also known as acetylsalicylic acid is a salicylate drug, often used as an analgesic to relieve minor aches and pains, as an antipyretic to reduce fever and as an anti-inflammatory medication. Chemically it is 2-acetoxybenzoic acid (Merck Index, 2001) and is a non steroidal anti-inflammatory drug and it is official in British pharmacopoeia (British Pharmacopoeia, 2004), Indian Pharmacopoeia (Indian Pharmacopoeia, 2007) and United States Pharmacopoeia (The United States Pharmacopoeia, 2006). Aspirin also has an antiplatelet effect by inhibiting the production of thromboxane, which under normal circumstances binds platelet molecules together to create a patch over damage of the walls within blood vessels. Because the platelet patch can become too large and also block blood flow locally and downstream, aspirin is also used long-term at low doses to prevent heart attacks, strokes, and blood clot formation in people at high risk for developing blood clots. It has also been established that low doses of aspirin may be given immediately after a heart attack to reduce the risk of another heart attack or of the death of cardiac tissue. Literature survey reveals that there are so many UV spectroscopic (Patel G.F, 2010),

HPLC methods (Nikelly J. G, 2006) and flurometric methods (Mohammad Mainul Karim, 2006) has been reported (Mishra P, 2006), (Purshotam K, 1964) for the pharmacokinetic study and pharmacological study of aspirin (Zenon Kokot, 1998). The present work aims to develop a simple, precise, accurate and validated difference spectroscopic method for the estimation of aspirin in tablet dosage form.

2. EXPERIMENTAL

2.1 Apparatus

UV-1700(E) 23 OCE, UV 1700 Pharmaspec, Shimadzu Corporation, with 1cm matched cell was used to carry out the UV detection. Commercially available aspirin tablets were procured from local market. The entire chemicals used were of AR grade.

2.2 Chemical and dosage forms

Freshly prepared 0.1M sodium hydroxide and 0.1M hydrochloric acid, 1M sodium salicylate and distilled water were used in the present study. The 1M sodium salicylate was prepared by weighing 1.60 gm of sodium salicylate and dissolving in 10 ml of fresh distilled water. Sodium salicylate is highly soluble in water and is used as hydrotropic agent. The 0.1M sodium hydroxide was prepared by weighing 400 mg of sodium hydroxide palates and then dissolving in 100 ml of water. The 0.1M hydrochloric acid was prepared by measuring 0.85 ml of concentrated hydrochloric acid and then dissolving in 100 ml of fresh distilled water.

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Received on: 23-09-2010

Revised on: 18-12-2010

Accepted on: 14-01-2011

Table 1: Calibration curve of proposed method

Sl.No.	concentration	Absorbance difference(n=6)	%RSD
1	0.1	0.125	0.66
2	0.2	0.222	0.92
3	0.3	0.410	0.05
4	0.4	0.59	0.64
5	0.5	0.76	0.79
6	0.6	0.89	0.18
7	0.7	1.04	0.78
8	0.8	1.24	0.88
9	0.9	1.39	1.01
10	1	1.55	0.88

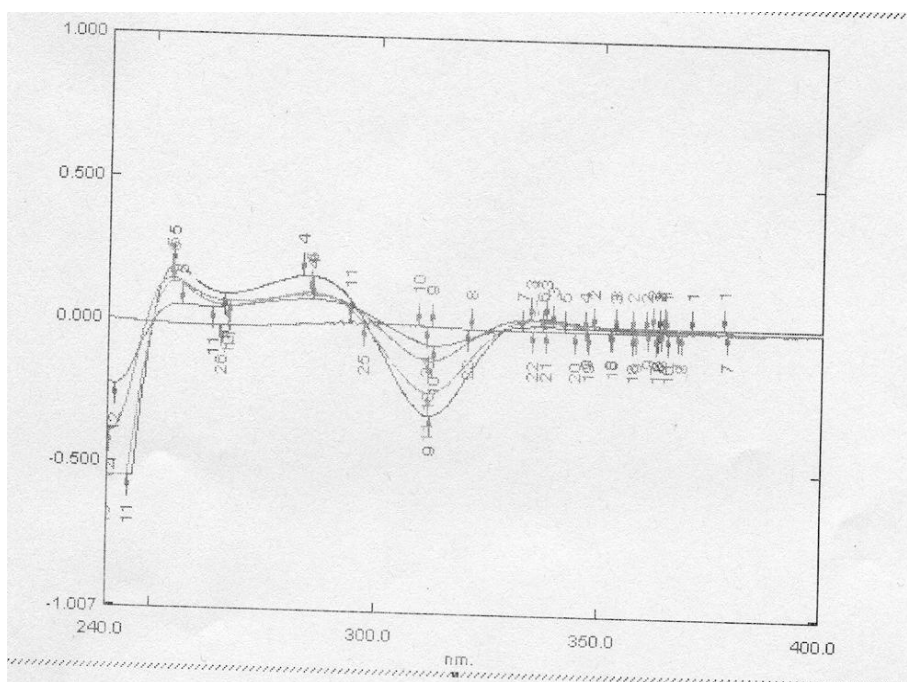


Figure 1: Difference spectrum of aspirin (different conc. of aspirin in 0.1 M HCl was taken as blank and the same concentration of drug in 0.1 M NaOH as sample)

2.3 Preparation of standard stock solution

50mg of aspirin was weighed accurately and dissolved in distilled water and made up the volume to 50ml in a volumetric flask with taking 1M sodium salicylate as hydrotropic agent. The solution was further diluted with 0.1M hydrochloric acid and 0.1M sodium hydroxide separately to get the concentration of 200 μ g/ml (working standard). Different aliquots were taken from their working standards and diluted with 0.1M hydrochloric acid and 0.1M sodium hydroxide separately to prepare a series of concentration from 0.1-1 μ g/ml as reference and test solution respectively. Difference spectrum was recorded by placing aspirin in 0.1M hydrochloric acid in reference cell and 0.1M sodium hydroxide in sample cell. Difference in absorbance between 312nm and 285nm was calculated to find out the amplitude. The calibration curve was prepared by plotting amplitude vs. concentration.

3. ASSAY OF THE MARKETED FORMULATION BY PROPOSED METHOD

Marketed tablet formulation was used for analysis. Twenty tablets were weighed accurately and their average net weight was calculated. The tablet were emptied and made to fine powder. The powder equivalent to 100mg of aspirin was weighed accurately and transferred in to 100ml volumetric flask. Dissolved in distilled water using sodium salicylate as hydrotropic agent. The solution was filtered through Whatmann filter paper no 41. From the stock solution 1 μ g/ml was prepared separately by using 0.1M hydrochloric acid and 0.1M sodium hydroxide. The amplitude was calculated by measuring the absorbance of equimolar concentration at maxima and minima in the difference spectrum. The amount of aspirin was calculated. The procedure was repeated for six times to perform precision (TABLE-2).

Table 2: Analysis of the tablet formulation

Sl.No.	Brand name of the tablet	Labeled amt. of drug	Amount found	% Label claim	Average	%RSD
1	Ecosprin	75	74.86	99.81	98.66	0.7236
2		75	75.28	100.37		
3		75	75.90	101.20		
4		75	74.20	98.93		
5		75	74.00	98.66		
6		75	75.32	100.42		

Table 3: Recovery study from drug solution

Amount of drug(μg) added to solution of pure drug	Recovery from drug solution	
	Mean (\pm SD) Amt(μg) found	Mean (\pm SD) % Recovery (n=6)
0.16	0.1594 (\pm) 0.61	99.62 (\pm) 0.32
0.20	0.1970 (\pm) 0.54	98.57 (\pm) 0.11
0.24	0.2386 (\pm) 0.7	99.41 (\pm) 0.051

4. RECOVERY STUDY

Accuracy of the proposed method was examined by recovery of the drug by standard addition technique. To the preanalysed formulation a known amount of the aspirin raw material was added in different concentration viz 80%, 100%, 120% in both reference and sample solutions. The procedure was repeated as per the analysis of formulation. The amplitude was calculated and the amount of aspirin recovered was determined. This was prepared for six times (TABLE-3).

5. RESULT AND DISCUSSION

A simple, precise and accurate difference spectrophotometric method has been developed for the estimation of aspirin in formulation. In this method the measured value is the difference in absorbance between two equimolar solutions of the analyte in different chemical forms which exhibit different spectral characteristics. The difference spectrum of aspirin in 0.1M NaOH was recorded by taking aspirin in 0.1M HCl as blank. The difference spectrum showed that the maxima at 312nm and minima at 285nm in alkaline solution drug shows more intense peak than acidic peak. Therefore ΔA is positive. Six point calibration graphs were constructed covering a concentration range 0.1- 1 $\mu\text{g}/\text{ml}$. Six independent determinations were performed at each concentration. Linear relationships between amplitude of maxima and minima of difference spectra versus the corresponding drug concentrations were observed. The standard deviation of the slope and the intercept were low. The correlation coefficient exceeded 0.999. The mean percent label claims estimated for the formulation was 98.66%. This value is very close to 100 indicating the accuracy of the proposed method. The low value of the statistical parameter viz standard deviation, percent co-efficient of variance validated the proposed analytical method. The mean percent recovery were within the range of 99.41 to 99.62%. Thus it is concluded that the proposed

method of analysis is new, simple, cost effective, environment friendly, accurate and reproducible and this method can be successfully employed in the routine analysis of aspirin in tablet formulation.

ACKNOWLEDGEMENT

The authors wish to thank Dr B.C.Roy College of Pharmacy, Durgapur for providing the necessary facilities to carry out the research work.

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