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Research Article

UV Spectrophotometric Method Development and Validation for Quantitative Estimation of Aspirin

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ABSTRACT

Aim: UV Spectrophotometric Method Development and Validation for quantitative estimation of Aspirin.**Objective:** U.V Spectrophotometric method have been widely employed for determination of analyte in a mixture. Our aim is to develop spectroscopic method for estimation of the Aspirin in ternary mixture by using U.V spectrophotometry. **Methodology:** The method was validated as per ICH guidelines. The recovery studies confirmed the accuracy and precision of the method. **Conclusion:** It was successfully applied for the analysis of the drug in bulk and could be effectively used for the routine analysis.

Key words: Aspirin, UV spectrophotometric method, Validation.

Introduction

Aspirin [2-acetoxybenzoic acid] is a nonsteroidal anti-inflammatory drug (NSAID) compound. Aspirin decomposes rapidly in solutions of ammonium acetate or the acetates, carbonates, citrates, or hydroxides of the alkali metals. It is stable in dry air, but gradually hydrolyses in contact with moisture to acetic and salicylic acids. In solution with alkalis, the hydrolysis proceeds rapidly and the clear solutions formed may consist entirely of acetate and salicylate. Aspirin, an acetyl derivative of salicylic acid, is a white, crystalline, weakly acidic substance, with a melting point of 136°C (277°F), and a boiling point of 140°C (284°F). Its acid dissociation constant (pK_a) is 3.5 at 25°C (77°F). The synthesis of aspirin is classified as an esterification reaction. Salicylic acid is treated with acetic anhydride, an acid derivative, causing a chemical reaction that turns salicylic acid's hydroxyl group into an ester group (R-OH → R-$OCOCH_3$). This process yields aspirin and acetic acid, which is considered a by-product of this reaction. Small amounts of sulphuric acid (and occasionally phosphoric acid) are almost always used as a catalyst (Bahadur et al., 2012; Savale et al., 2017).

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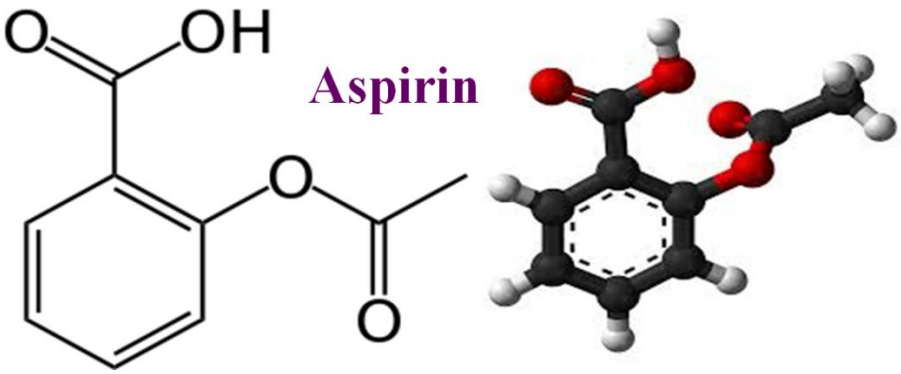

Material and Method

Material

Aspirinsupplied as a gift sample by Loba chem. Pvt. Ltd (Mumbai, India) used as working standard.

Instrumentation

A double beam UV-VIS spectrophotometer (UV-1700, Shimadzu, Japan) connected to a computer loaded with spectra manager software UV Probe was used. The spectra were obtained with the instrumental parameters as follows: Wavelength range: 200–400 nm. All weights were taken on an electronic balance (Model Shimadzu AUX 120).

Preparation of standard stock solution

According to European pharmacopoeia, 10 mg of aspirinwas dissolve in 100 ml of methanol (100 µg/mL). Out of this stock 0.2-1.2 ml was pipetted and diluted up to 10 ml by methanol (2-12 µg/mL) and examined between 200-400 nm. The maximum absorbance was determined using UV-Vis Specrophotometer (UV-1700, Shimadzu, Japan) to confirm the λmax of the drugs (Savale et al., 2017).

Validation of analytical method

The analytical performance characteristics which may be tested during methods validation: % Recovery, Precision, Ruggedness and sensitivity (Kuchekar et al., 2003; Savale et al., 2017).

Results and Discussion

Method Development

The solution of aspirinin methanol was found to exhibit **maximum absorption at 243 nm** after scanning on the UV-Vis spectrophotometer which was reported as λmax in the literature and the procured drug sample of aspirincomplies with the reference spectra (Figure 1).

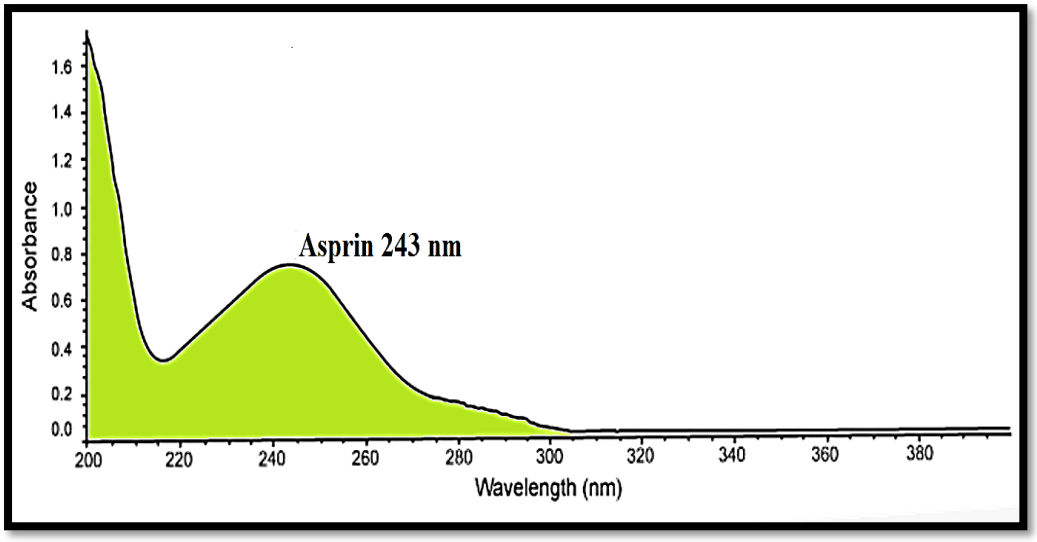

Figure 1. UV spectra of Aspirin

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Validation of analytical method

Linearity

Accurately weighted aspirin(10 mg) was dissolved in 100 ml of methanol to obtain working standard of 100 µg/ml. Aliquots were pipetted from the stock solution of drug and were transferred to 10 ml volumetric flask, the final volume was adjusted with methanol so that concentration of 2-12 µg/ml could be made. Absorbance of the above solution were taken at 257 nm by using UV-Vis spectrophotometer (UV-1700, Shimadzu, Japan) against the blank solution prepared in the same manner without adding the drug. A graph of absorbance vs concentration was plotted (Figure 2) and R^2 was found to be 0.9999.

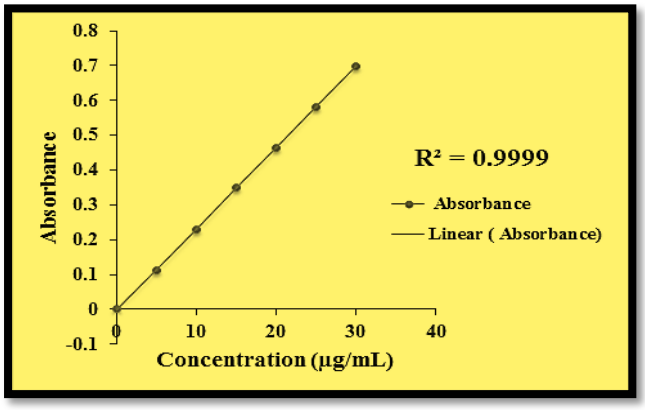

Figure 2. Calibration curve of Aspirin

Recovery

Recovery study is performed by standard addition method by adding the known amount of aspirin(Working standard) at two different concentration levels i.e 80%, 100% of assay concentration and % recovery for all these drug were calculated. Result was reported in Table 1.

Table 1. Recovery study

Drug	Initial amount (µg/ml)	Added Amount (µg/ml)	% Recovery	% RSD (n = 3)
Aspirin	2	1.8	100.85	0.01
	2	2	100.03	0.04

Precision

Intra-day precision was determined by analysing, the two different concentrations 2 mg/ml, 3 mg/ml containing aspirin, for three times in the same day (n = 3) Table 2. Inter-day variability was assessed using above mentioned three concentrations analysed on three different days, over a period of one week (n = 3) Table 2.

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Table 2. Presion study

		Intra - Day		Inter - Day	
Drug	**Con. (µg/ml)**	**Mean ± SD**	**% RSD**	**Mean ± SD**	**% RSD**
Aspirin	2	2.0 ± 0.0086	0.01	2.0 ± 0.0059	0.04
	3	3.0 ± 0.0093	0.05	3.0 ± 0.0073	0.07

Ruggedness

From stock solution, sample solution containing aspirin(2 µg/ml) was prepared and analyzed by two different analysts using similar operational and environmental conditions (Table 3) (n = 3).

Table 3. Ruggedness study

	% Amount Found		% RSD	
Drug	**Analyst I**	**Analyst II**	**Analyst I**	**Analyst II**
Aspirin	100.00	100.45	0.04	0.01

Sensitivity

Sensitivity of the proposed method were estimated in terms of Limit of Detection (LOD) and Limit of Quantitation (LOQ) (Table 4).

Table 4. Sensitivity study

Drug	**LOD**	**LOQ**
Aspirin	0.58 ± 0.004	0.99 ± 0.015

Conclusion

The proposed UV spectrophotometric method was found very simple, rapid and economical. The method is validated in compliance with ICH guidelines is suitable for estimation of Aspirinwith excellent recovery, precision and linearity.

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