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## DEVELOPMENT AND VALIDATION OF UV SPECTROPHOTOMETRIC METHODS FOR ESTIMATION OF ALFUZOSIN IN BULK AND PHARMACEUTICAL FORMULATION

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### ABSTRACT

Three simple, economical, precise and accurate UV spectrophotometric methods have been developed for the estimation of alfuzosin in bulk and pharmaceutical formulations. Method A involves measurement of absorbance of solution at 244.99 nm in methanol. Method B involves integrating area under curve between the wavelength ranges of 243.34 nm – 246.63 nm and is used to construct calibration curve. Method C is first order derivative spectroscopy method, in which first derivative amplitude at 235.12 nm is measured. Linearity for detector response was observed in the concentration range of 2.5-20 µg/ml for all the three methods in methanol. The results of analysis have been validated as per ICH analytical method validation guidelines. Accuracy, precision and recovery studies were carried and confirmed the accuracy of the proposed methods.

**Keywords:** Alfuzosin, UV - Spectrophotometric methods, area under curve.

### INTRODUCTION

Alfuzosin is chemically known as N-[3-[(4-amino-6,7-dimethoxy-quinazolin-2-yl)-methyl amino] propyl] tetrahydrofuran-2- carboxamide (Fig. 1). Alfuzosin is alpha-adrenergic blockers <sup>[1]</sup> and

relaxes the muscles in the prostate and bladder neck, making it easier to urinate. Alfuzosin is used to improve urination in men with benign prostatic hyperplasia.<sup>[2]</sup>

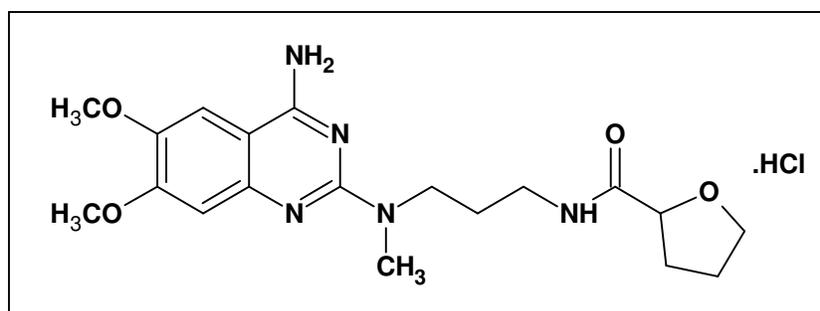
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**Figure: 1**-Chemical structure of Alfuzosin.

Several analytical methods that have been reported for determination of alfuzosin biological fluids and pharmaceutical formulation which includes RP-HPLC method [3, 4], HPTLC methods<sup>[5]</sup>, validated HPLC and HPTLC stability-indicating methods<sup>[6]</sup>, RP-HPLC method for simultaneous estimation of Alfuzosin Hydrochloride and Dutasteride<sup>[7]</sup>. There is no simple and economic UV Spectrophotometry method available for its determination. Therefore aim of the study was to develop and validate simple UV absorbance, area under curve and derivative spectrophotometric methods for estimation of alfuzosin in bulk and formulations.

#### **OBJECTIVE**

The aim of present work is to develop and validate simple, sensitive, specific, spectrophotometric methods for the detection of alfuzosin in bulk and pharmaceutical formulation

#### **MATERIALS AND METHODS**

##### **Instrumentation**

An UV-Visible double beam spectrophotometer of makes Varian Cary 100 with 10 mm matched quartz cells was used. All weighing were done on electronic balance (Model Shimadzu AUV-220D).

##### **Reagents and chemicals**

Pure drug sample of alfuzosin (% purity- 99.8) was kindly supplied as a gift sample by Dr.Redddy's lab Ltd. Tablet used for analysis was alfoo (Dr. Redddy's lab Ltd. (Batch no-B91887) (Formulation T1) and alfusin (Cipla (Batch no-M1201) (Formulation T2) containing alfuzosin equivalent to 10 mg of alfuzosin hydrochloride.

Spectroscopic grade methanol was used throughout the study.

##### **Preparation of standard stock solution**

An accurately weighed quantity about 10 mg of alfuzosin was transferred to 100 ml volumetric flask; it was dissolved in sufficient quantity of methanol. The solution was further diluted with methanol to get the suitable concentration range.

##### **Preparation of calibration graph**

Standard stock solution was suitably diluted with methanol to get concentration in the range 2.5 - 20 µg/ml and linearity was studied by using zero absorbance method (method A) by measuring absorbance at 244.99 nm, by area under curve method (method B) by using integrated between the wavelengths 243.34 - 246.63 nm and by measuring first derivative amplitude at 235.121 nm (method C).

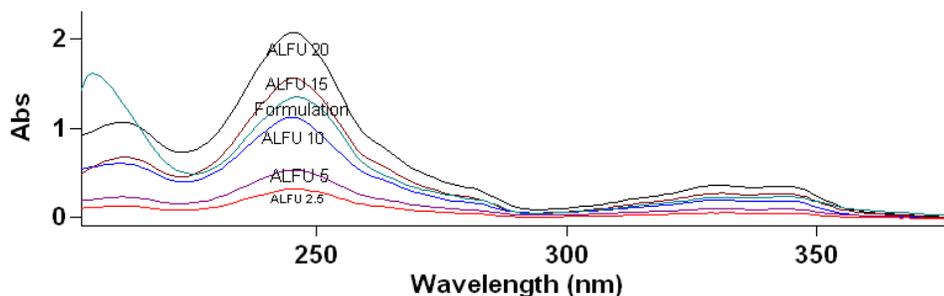
##### **Formulation analysis**

Twenty tablets were weighed accurately and crushed to fine powder. Quantity of tablet powder equivalent to 50 mg of analyte was weighed and transferred to 50 ml volumetric flask and dissolved in 40 ml of methanol. This solution was then filtered through Whatmann filter paper no. 41. The volume was made up to 50 ml with methanol by washing the filter and proposed methods were followed.

#### **METHOD DEVELOPMENT** [8-12]

##### **Absorbance method (Method A)**

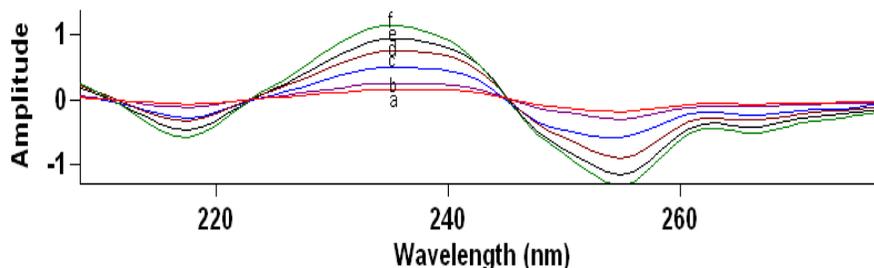
The method involves measurement of absorbance of solutions in the concentration range of 2.5-20 µg/ml at 244.99 nm (Fig.2).



**Figure: 2** - Overlay spectra of alfuzosin standard (2.5 – 20 µg/ml) and formulation (12.5 µg/ml) in methanol

**Area under curve method (Method B)**  
 The AUC (Area under Curve) method involves the calculation of integrated value of absorbance with respect to the wavelength between two selected wavelength 243.34 nm and 246.63 nm (Fig.2).

**The first order derivative method (Method C)**  
 The first order derivative spectra of Alfuzosin solution show a sharp peak and linearity at 235.121 nm (Fig.3)



**Figure: 3** -First order derivative spectra of alfuzosin standard solution and extracted from formulation as shown in Fig. 2.

**Precision**

The precision of the assay was determined by repeatability and intermediate precision (intraday and inter-day) and reported as % RSD. For this 5

µg/ml, 7.5 µg/ml and 12.5 µg/ml concentration of solution was analysed three times in day and similarly it was analysed on three days and the % RSD values were calculated. (Table 1)

**Table 1-Optical Characteristics and validation data of alfuzosin**

Parameter		Method A	Method B	Method C
λ (nm)		244.99 nm	243.34 – 246.63 nm	235.12 nm
Beer's law limit (µg/ml)		2.5-20 µg/ml	2.5-20 µg/ml	2.5-20 µg/ml
Regression Equation (y = mx + c)	Slope (m)	0.1039024	0.339878	0.0471008
	Intercept (c)	0.0090244	0.0612805	0.0148644
	Correlation coefficient	0.9988	0.9988	0.999
Precision (%R.S.D.) n=6	Repeatability	0.65	0.54	0.79
	Intra-day	0.59	0.69	0.62
	Inter-day	0.54	0.62	0.95
Tablet Assay (%), % RSD	Formulation I	99.32, 0.78	99.57, 0.71	98.77, 0.67
	Formulation II	99.12, 0.73	98.54, 0.69	99.34, 0.74
Method sensitivity	LOD (µg/ml)	0.158	0.167	0.217
	LOQ (µg/ml)	0.481	0.509	0.658

**Recovery**

The accuracy of the method was evaluated through standard addition method. In this known amount

of standard alfuzosin was added in pre-analyzed sample. This was done for 2.5 µg/ml, 5µg/ml, and 7.5 µg/ml and in triplicate. (Table 2)

**Table 2-**Recovery study of Alfuzosin by using proposed methods.

Recovery Level	Amount Spiked in (µg/mL)	% Mean Recovery, % RSD by (n = 6)		
		Method A	Method B	Method C
50%	2.5	100.95, 0.50	99.72,0.75	99.87, 0.70
100%	5	99.91, 0.43	96.89, 0.87	100.57, 0.97
150%	7.5	99.73, 1.24	99.92 ,0.47	98.37, 0.87

**RESULT AND DISCUSSION****Method validation**<sup>(13, 14)</sup>:

Using appropriate dilutions of standard stock solution, the solutions were scanned. The zero order overlain spectra are shown in Fig.2. A critical evaluation of proposed method was performed by statistical analysis of data where slope, intercept, correlation coefficient is shown in Table - 1. As per the ICH guidelines, the method validation parameters checked were linearity, accuracy, and precision and method sensitivity

Beer's law obeyed in the concentration range (2.5 - 20 µg/ml) and with correlation coefficient > 0.9999 for the proposed methods. The proposed method was also evaluated by the assay of commercially available tablets containing Alfuzosin hydrochloride

The % assay was found to be in the range of 98.54% - 99.57% for both the formulations by all the three proposed methods as shown in Table-1. Results of recovery studies are presented in Table-2. For, alfuzosin the recovery study results ranged from 98.37 % to 100.95 % with % RSD values ranging from 0.43 % to 1.24 %. The accuracy and reproducibility is evident from the data as results are close to 100% and standard deviation is low. Result of precision and recovery shows that the methods are precise and accurate.

**CONCLUSION**

The proposed UV Spectrophotometric methods for the determination of alfuzosin in bulk and pharmaceutical formulation are very simple, precise and accurate. Therefore the methods can be used as routine IPQC test for bulk and tablet formulations of the analyte.

The validated spectrophotometric methods employed here proved to be simple, fast, accurate and precise and sensitive thus can be used for routine analysis of Alfuzosin.

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