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(RESEARCH ARTICLE)

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Analytical method development and validation of Alfuzosion and Dutasteride by RPHPLC

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Abstract

The Aim is Development & Validation of Alfuzosin 10Mg & DutaSteride 0.5mg Using Parameters such Precision Accuracy, linearity, specificity, robustness, Ruggedness.

The proposed project success full attempt has been made to develop simple accurate economic & rapid methods for the estimation of tablet formulation & to validate the methods as a result three simple accurate methods were developed & validated.

The System Suitability Parameters Prove that the Proposed method is equally suitable for estimation of ALFU& DUTA the Chromatogram For Sample were found to be Satisfactory on Rp-18(2)250*4.6mm 5µm column using mobile phase combination of buffer Acetonitrile (58;42v/v) with flow rate of 1.0ml/min both the peaks were found to be symmetrical as found from symmetry from of 1.01 for ALFU &DUTA .resolution proposed method satisfactory baseline peak showing separation .Retention time Duta-5min and aifu-2min estimate theoretical plate 5689 per meter recovery of drug acceptance limit 97-103 % .Runtime 8min detector used UV detector 225nm. flow rate 1.0 ml /min the method as found liner over the concentration 20-120mg/ml for ALFU& 1-6mg /ml for DUTA correlation coefficient of0. 998 the method has been validated as per the guideline given by ICH requirements to assure that the method consistently meets the Predetermined Specifications & quality attributes

Keywords ALFU; DUTA; Chromatogram; RP-HPLC; Quality attributes

1. Introduction

1.1. RP HPLC

Reversed-phase high performance liquid chromatography (RP-HPLC) is a technique that separates compounds based in their polarity

1.2. Analytical method development and validation of Alfuzosin and dutasteride by RPHPLC

Standard analytical procedure for newer drugs of formulation may not be available in pharmacopoeias hence it is essential to develop newer analytical methods which are simple ,accurate , precise ,specific economic ,linear & rapid from the literature review it was found that a very few analytical methods have been reported for the simultaneous estimation of Alfuzosin & Dutasteride by RPHPLC. Chemically Alfuzosin chemical name N(3-(c4-amino-6,7-di methoxy –quinazolin -2-yl)methyl amino propyl tetra hydro furan-2 carboxamide. category anti-hypertensive MOA adrenergic alpha antagonists .A white crystalline power & highly hygroscopic alpha(1) adrenergic blocking agent that exhibits selectivity for alpha (1) adrenergic receptors in the lower urinary tract the relaxation of smooth muscle bladder neck &

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prostate resulting urine &flow reduction in symptoms prostate hyperplasia ADR dizziness fatigue .Dutasteride chemical name (5a17b) –N- (2.5bis (triflurомethyl)phenyl-3- OXO-4azxan dross -1-rne -17-carboxamide category enzyme inbitor MOA dual 5-alpha reductase inhibitor that inhibit version of testo sterone to dihydro testrsterone inhibits both iso forms ADR decrease libido ejaculation disorder

2. Literature Review

Literature survey reveals that analytical & bio analytical methods have been reported HPLC assay validated alfuzosin HCL in HPLC .and RPHPLC bulk dosage form and spectrometer method absorbance. Dutastride sample HPTLC bulk & tablet dosage from validation novel a at LC-MS development validation method estimate human plasma.

3. Materials and Methods

ALFU & DUTA procured as gift samples Chandra labs pvt.ltd Hyderabad pharmaceutical dosage from containing 10mg ALFU&0.2 DUTA product of cipla was purchased from local drug store acetonitrile and water of HPLC grade potassiumdihydrogenphosphate .sodium dihydrogen phosphate of AR grade was purchased from Merck, Mumbai.

3.1. Instrument

The chromatographic analysis was performed shimadzu separation module Lc-20at prominence liquid chromatography a HPLC waters alliance 2695 separation module with UV equipped with empower software for data processing the chromatographic optimized method column kromasil c18,150*4.6mm 5m universal column washed.

3.2. Chromatographic condition

Mobile phase consisting of aceto nitrile: mixed phosphate buffer (65:35) was used in isocratic mode with UV detector 225nm at 25c. An injection volume of 20µl was used keeping the flow rate at 1.0ml/min the Retention times for Alf & Duta under the optimized chromatographic condition was found to be 8min.

3.3. Preparation of buffer

Take 1.034 gm. of potassium dihydrogen phosphate (0.02m) .0.190gm of dipotassium hydrogen ortho phosphate (0.003m) dissolve in 350ml HPLC water and sonicated for 10min filtered in 0.25microns in membrane filter

3.4. Preparation of mobile phase

The preparation of mobile phase filtered and degassed mixture of Acetonitrile and buffer in the ratio of 65:35 and filter through 0.25 micron membrane filter.

3.5. Preparation of standard solution

Accurately weighed quantity of 2.5 mg Duta & 50mg Alf was transferred into 50ml Volumetric flask. Dissolve in about 10ml methanol of mobile phase sonic ate about 10minuntil all the content has been dissolved and make up volume with mobile phase the concentration of ALFU &DUTA were found to be 50µg/ml and 1000µg/ml.

3.6. Preparation of sample solution

Weigh about 10 tablet and powder form that an equivalent amount of 0.5 mg of DUTA &10mg of ALFU was taken into 25ml volumetric flask. Add about 10ml of methanol and sonic ate about 10min until all the content has been dissolved volume makeup mobile phase filter used 0.25µ membrane filter under vacuum. Volume mark made up mobile phase.

3.7. Assay

An accurately weighed quantity of 50mg ALFU&2.5mg of DUTA was transferred in 100ml volumetric flask dissolved in methanol and sonicated about 10min until all the content has been dissolved volume make up mobile phase filter used 0.25µ membrane filter vacuum inject 20µof sample measure the of sample calculate percentage of assay.

4. Result and Discussion

By using the optimized chromatographic condition the method was validated for linearity, accuracy, precision, specificity, robustness, and ruggedness by the following procedure as per ICH guide lines.

4.1. Precision

The precision data of the system. the %RSD for Alf and Duta Were found to be 0.65 and 0.24 respectively. Hence the precision of the system as found to be well in the acceptance criteria (Not less than 2%)

4.2. Linearity

The linearity standard solution concentration 20-120µg/ml of ALFU and 1-6ug/ml of DUTA the relationship between the concentration and the peak response of ALFU was linear in specific range the regression coefficient was found to be 0.998.

4.3. System suitability

Standard solution of DUTA & ALFU was determined under proposed condition chromatogram indicating satisfactory %RSD of peak responses theoretical plate asymmetry and retention time

Theoretical plate not less than 2000, retention time not less than 2 , Asymmetry not less than 2.

4.4. Specificity

The specificity performed various degradation products are formed and there is no change in the detection of the analytic in the presence of other components no interference at the rention time of DUTA and ALFU

4.5. Accuracy

The standard solution to 100ml mobile phase the concentration of solution becomes 120mcg of ALFU and 6mcg DUTA 1ml of spikiness standard recovery % of 99.59.

4.6. Spiking standard

The standard solution of 10ml of stock solution diluted with 100ml with mobile phase average 241.75 and mcg recovery 6.473 and %recovery 99.59.

4.7. Robustness

The standard solution sample 202.3mg 25ml volumetric flask 5ml pipette out 0.25µ membrane filter flow rate 0.9ml/min wave length 223-227nm when wave length was changed there was change in the RT when temperature and PH were changed there no changes observed. While change all the above parameters the assay % was observed to be within limit

4.8. Ruggedness

The standard solution 100mcg was injected for by different analyst the area for inject in HPLC was measured the %RSD for the area of replicate inject was found to be within the specified limit less than 0.1

Table 1 Validation parameter of the HPLC method for alfuzosin HCL and dutasteride

S.no	Validation parameter	Alfuzosin hcl	Dutasteride
1.	Linearity(µg/ml)	20-120µg/ml	1-6 µg/ml
2.	Correlation co effient	0.998	0.998
3.	Precision(%RSD)	0.65	0.24
4.	Tablet Assay (%)	99.47	99.97
5.	Theoretical plate(N)	4489	7921
6.	Tailing factor	1.7	1.4

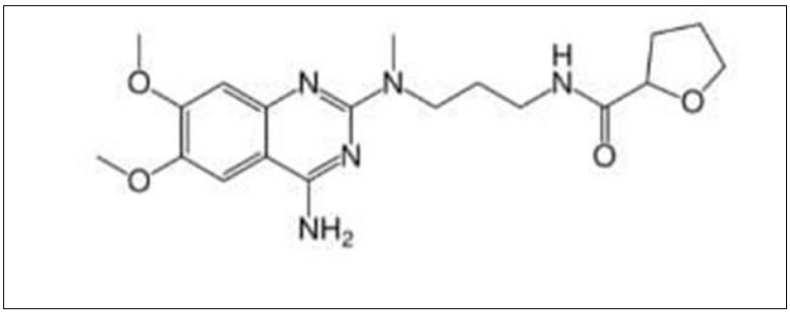


Figure 1 Chemical Structure of Alfuzosin

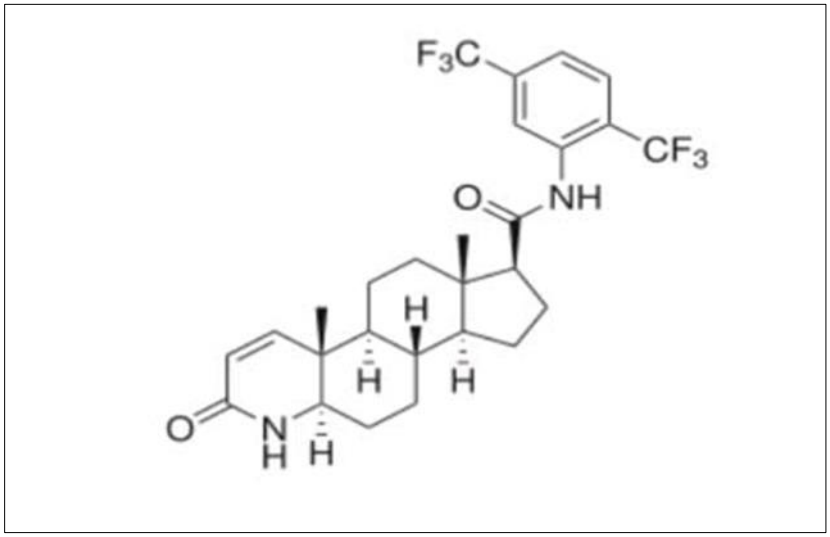


Figure 2 Chemical Structure Of Dutasteride

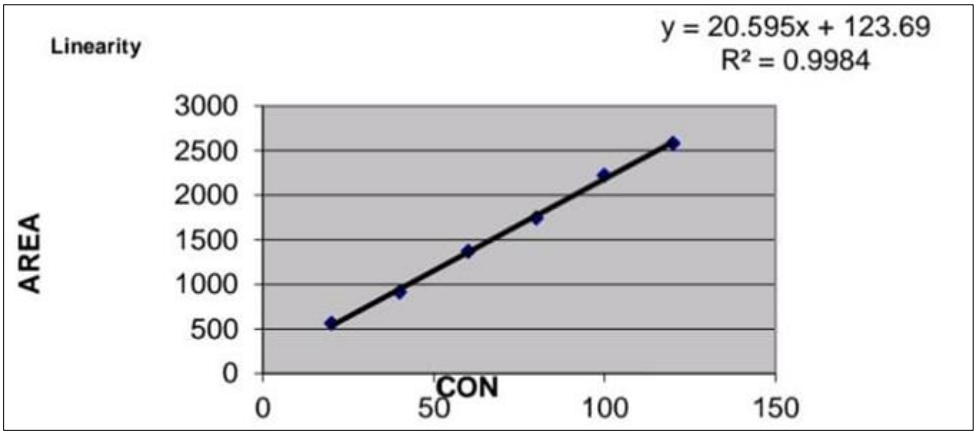


Figure 3 Calibration Curve of Alfuzosin

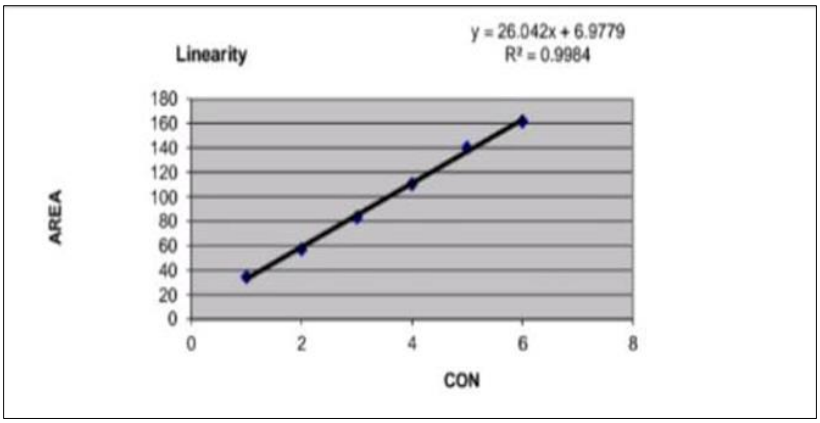


Figure 4 Calibration Curve of Dutasteride

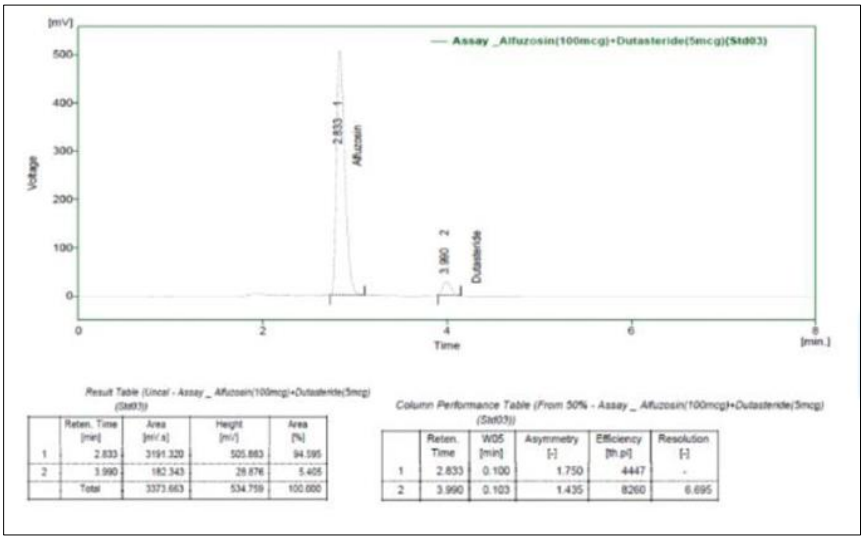
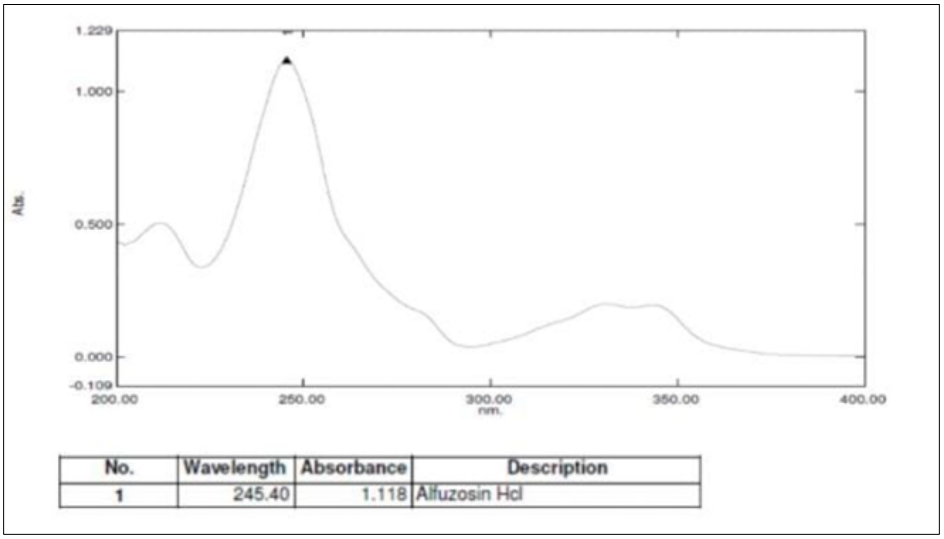


Figure 5 Assay of Alfuzosin 100mcg &DUTASTERIDE 5mcg



No.	Wavelength	Absorbance	Description
1	245.40	1.118	Alfuzosin Hcl

Figure 6 UV Spectrum of Alfuzosin HCL

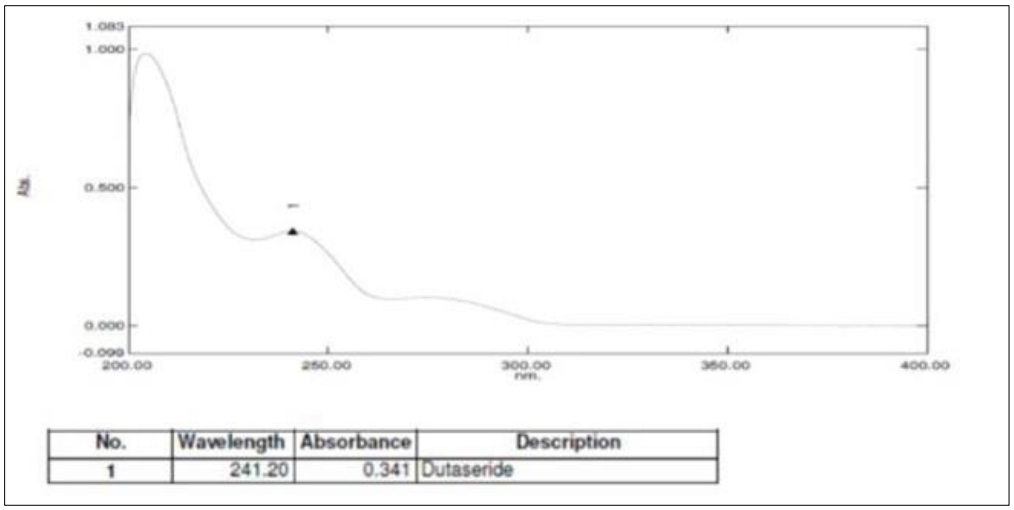


Figure 7 UV Spectrum of Dutaseride

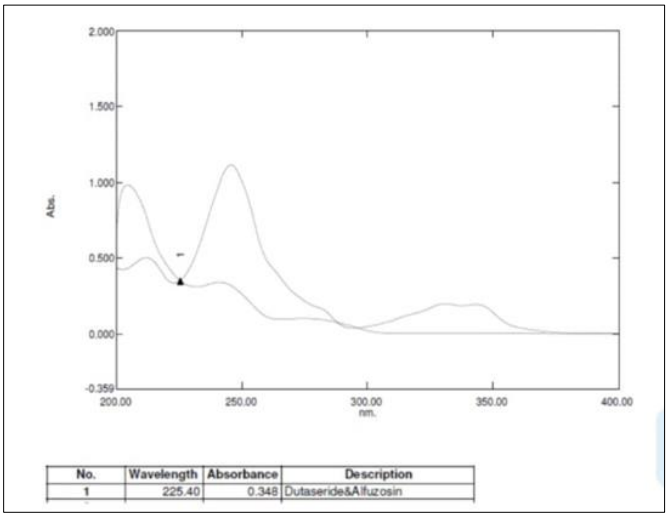


Figure 8 Overaid UV Spectrum Of Alfuzosin And Dutaseride

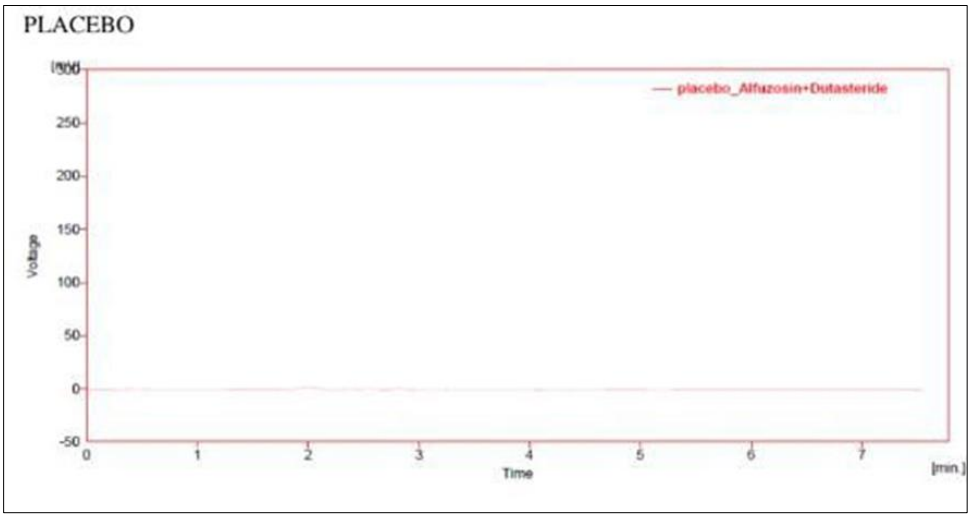
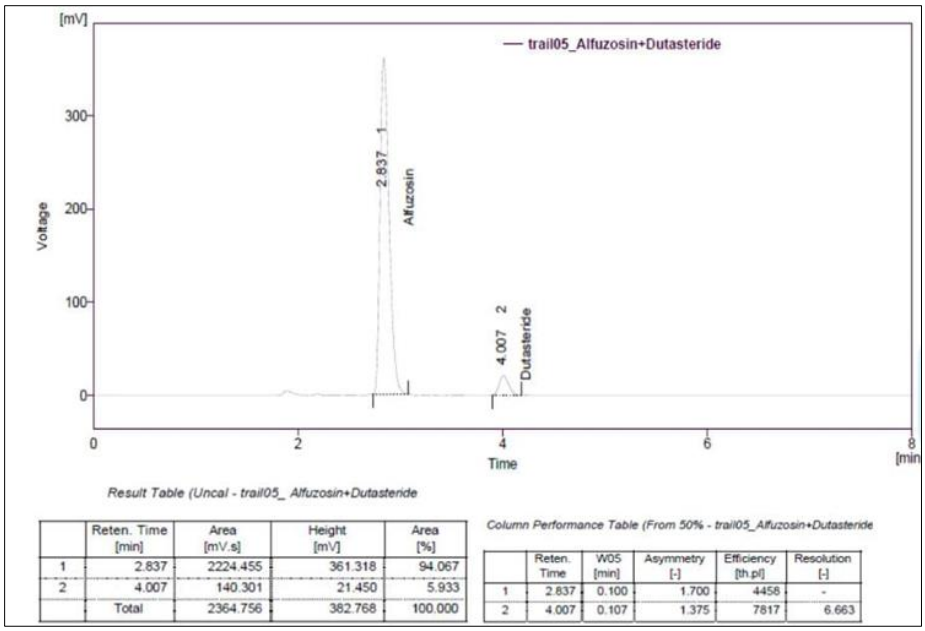


Figure 9 Placebo



Result Table (Uncal - trail05_ Alfuzosin+Dutasteride

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]
1	2.837	2224.455	361.318	94.067
2	4.007	140.301	21.450	5.933
	Total	2364.756	382.768	100.000

Column Performance Table (From 50% - trail05_Alfuzosin+Dutasteride

	Reten. Time	W05 [min]	Asymmetry [-]	Efficiency [th.pl]	Resolution [-]
1	2.837	0.100	1.700	4458	-
2	4.007	0.107	1.375	7817	6.663

Figure 10 Optimized Chromatography method Of Alfuzosin & Dutasteride

5. Conclusion

The RP HPLC method was developed and validated successful for simultaneous estimation of ALFU & DUTA in tablet dosage form. the present study was validated as per the ICH guidelines and the method was found to be accurate precise , linear , specific , reproducible for the simultaneous ..This Study extended by studying the degradation kinetic of ALFU&DUTA determination by RPHPLC method and also its estimation in plasma and biological fluids.

Compliance with ethical standards

Disclosure of conflict of interest

No conflict of interest to be disclosed.

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