Zero and First Order Derivative Method Development and Validation of Silodosine Bulk and Its Pharmaceutical Formulation by Using UV-Visible Spectroscopy

[1]Mis.Snehal S. Giri, [2]Mr.Amiksh K. Pukale, [3]Mr.Santosh A.Waghmare, [4]Ms. Pratibha Kale, [5]Ms. Snehal Jadhav
[1]Student, [2]Student, [3]Guide, [4]Student, [5]Student
[1]M. Pharmacy (QAT),
[1]JSPM's Charak College of Pharmacy and Research, Wagholi, Pune, India

Abstract: A simple, sensitive, rapid and reproducible UV method has been developed and validated for Calibration determination of Silodosine in bulk and in pharmaceutical formulation. For development of UV method for Standard stock solutions of Silodosine are prepared by using methanol and further dilutions for sample are prepared by using distilled water. Quantitative method development by zero order derivative method and first order derivative method was measured at 266 and 325nm respectively. Standard curve showed a regression coefficient zero order derivative and first order derivative is 0.999 and 0.998 respectively. The method was validated as per ICH guidelines. The precision and repeatability results showed % RSD less than 2%.

***Keywords*- UV, Silodosine, Zero order, First order.**

I. INTRODUCTION

Silodosin is a highly selective alpha 1A-adrenoreceptor antagonist approved for the treatment of the signs and symptoms of benign prostatic hyperplasia. Its clinical pharmacology profile offers a number of advantages including uroselectivity, once daily (QD) dosing, a standard dose of 8 mg QD that does not need to be adjusted according to age and the feasibility of concomitant treatment with phosphodiesterase type 5 inhibitors and antihypertensive agents. Relative to Tamsulosin, Silodosin has less cardiovascular side effects.

Silodosin, a selective antagonist of alpha-1adrenoreceptors, has chemical name 1-(3-Hydroxypropyl)-5-[(2R)-2-({2- [2-(2,2,2-trifluoroethoxy)phenoxy]ethyl}amino)propyl]-2,3-dihydro-1H-indole-7-carboxamide and the molecular formula is C25H32F3N3O4 with a molecular weight of 495.53(Fig. 1).

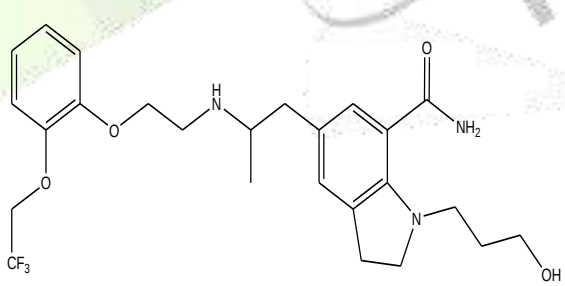

Fig. 1 Chemical Structure of Silodosine

The structural formula of Silodosin is: Literature survey reveals that number of analytical methods are available for estimation of Doxazosin, Tamsulosin, Gabapentine and other BPH drugs but only one UV spectrophotemetric method and one HPLC method has been developed for the quantitative estimation of Silodosin in formulation and one LC-MS/MS method for the determination of Silodosin in human plasma. Silodosin is a key drug for the treatment of BPH with a number of advantages including uroselectivity, once daily dosing, standard dose of 8 mg QD that does not need to be adjusted according to age and the feasibility of concomitant treatment with phosphodiasterase type inhibitors and antihypertensive agents. Lack of analytical methods for the quantitative estimation drives us for the development of spectrophotometric methods for the routine analysis of Silodosin.

II. EXPERIMENTAL:

2.1 Reagents and chemicals:
Silodosine API
Silodosine (Pharmaceutical Formulation): Silodosine TM 4mg, MSN Laboratories PVT LTD

2.2 Apparatus:
Volumetric Flask, Beaker, Pipette, Funnel.

2.3 Instrument:
Shimadzu 1800 UV-Visible spectrometric

2.4 Stock solution preparation:
Accurately weighed 10mg silodosine bulk drug take in a 10ml volumetric flask, dissolved in sufficient amount of the methanol as solvent and make up with it. transfer this all 10ml solution in to a 100ml volumetric flask and make up with the distilled water. which is a 100ug/ml.

2.5 Sample Preparation:
Withdraw 1 ml to 5 ml from 100ug/ml stock solution in 10ml volumentric flask and volume make up with distilled water to make 10ug/ml to 50 ug/ml respectively.

2.6 Zero order method:
The standard stock solution of the different concentrations is scan in the range of at 200-400nm and absorbance were recorded for 10-40 ug/ml at λ max 266nm. From this calibration curve is (fig. 2) plotted as follow:

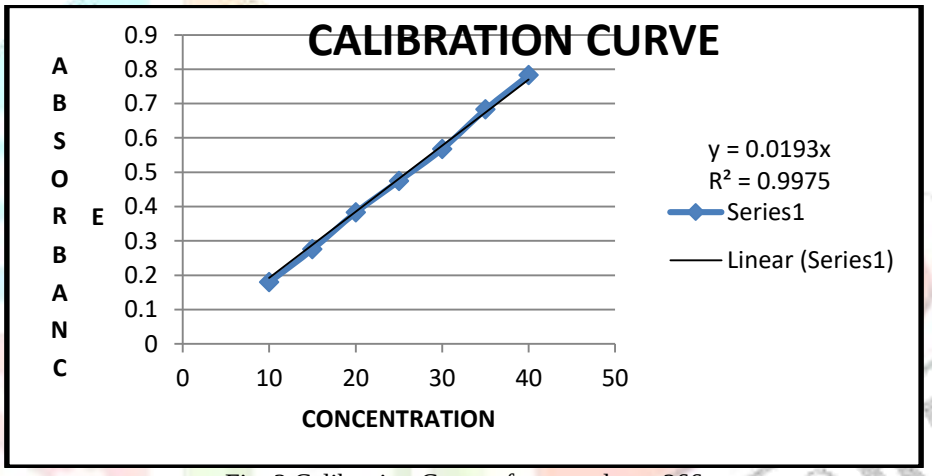

Fig. 2 Calibration Curve of zero order at 266

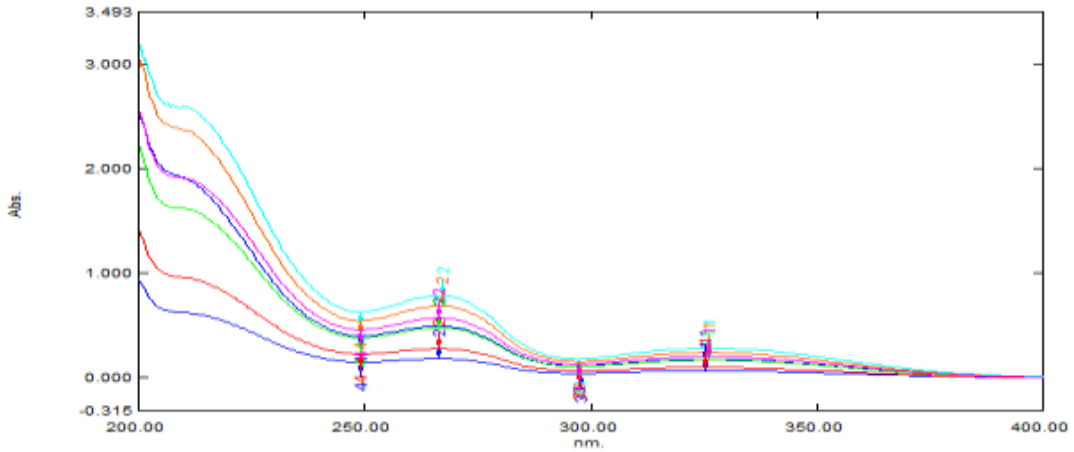

Fig. 3 Overlay of Zero order at 266

2.7 First order method:
The standard stock solution of the different concentrations is scan in the range of at 200-400nm and absorbance were recorded for 10-50 ug/ml at λ max 325nm. From this calibration curve is (fig no.4) plotted as follow:

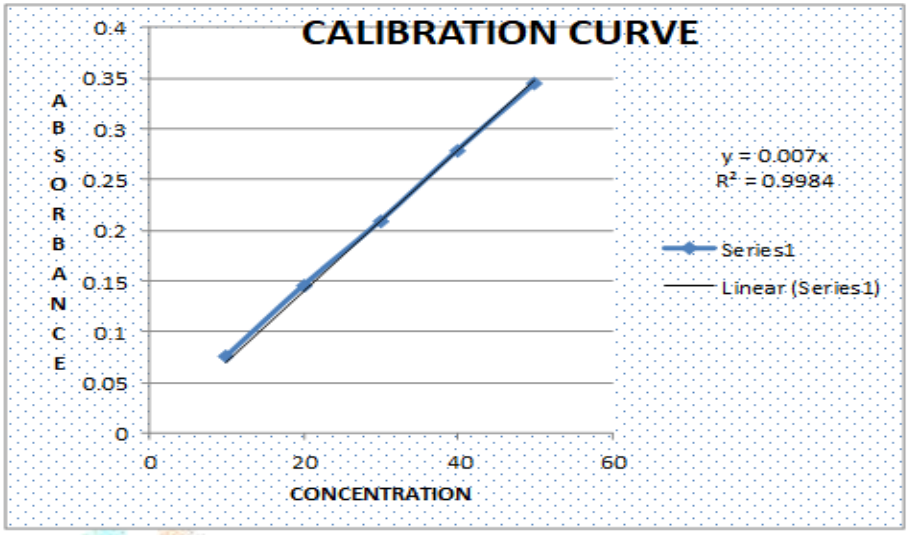

Fig. 4 Calibration Curve of First order at 325

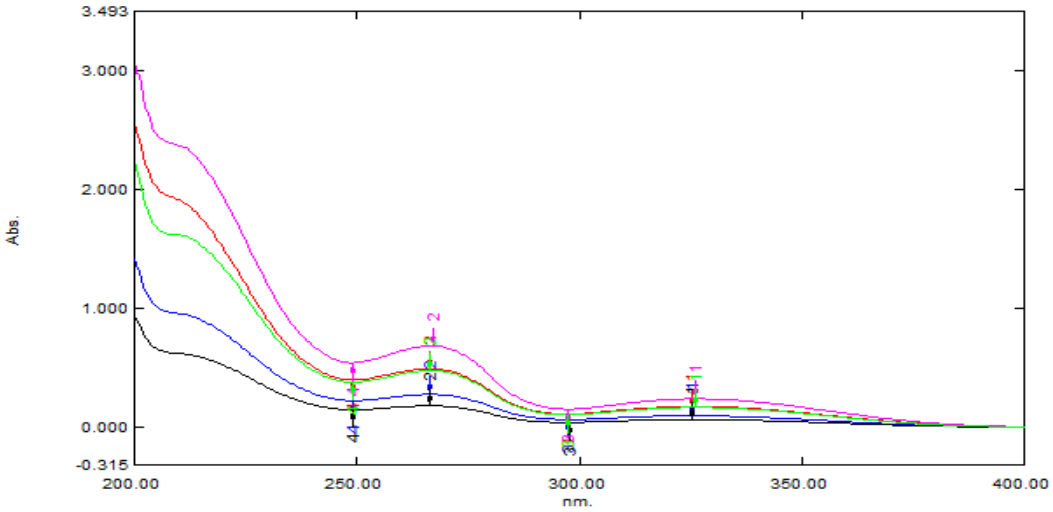

Fig. 5 Overlay of first order at 325

III. Results and Discussion:

The zero order and first order derivative spectra for Silodosine were recorded at the wavelength of 266 nm and 325 nm respectively.

1.Linearity:

The linearity was observed in the range of 10-40ug/ml and 10-50ug/ml for zero order and first order respectively and regression coefficient found to be 0.9975 and 0.984 for zero order and first order respectively.

2.Precision:

The precision of an analytical procedure expresses the closeness of between the series of measurements obtained from multiple sampling of the same homogeneous sample under the prescribed conditions. The 6 concentrations of 10ug/ml at 266nm and 325ug/ml for interday and intraday precision for zero order and first order method respectively. Results obtained are shown in table 1.

Table 1: Interday and Intraday precision for Zero order at 266nm

	Precision Interday				Precision Intraday		
	0 min	**1 hr**	**2 hr**		**1 day**	**2 days**	**3 days**
SD	0.003606	0.004041	0.005132	**Sd**	0.003512	0.003	0.003606
RSD%	1.102615	1.24608	1.596971	**rsd**	1.707561	1.522843	1.868161

Table 2: Interday and intraday precision for First order method at 325

	Precision Interday				Precision Intreday		
	0 min	**1 hr**	**2 hr**		**1 DAY**	**2 DAY**	**3 DAY**
SD	0.003055	0.003512	0.003606	**SD**	0.002082	0.001155	0.001155
RSD%	1.353789	1.572486	1.602467	**RSD%**	1.469411	0.897436	0.859579

3.Robustness:

Robustness is a measure of capacity of a method to remain unaffected by small, but deliberate variations in the method conditions, and is indications of the reliability of the method.A method is robust, if it is unaffected by small changes in operating conditions.Prepare 6 concentrations of 10 ug/ml by water and methanol.result obtain is shown in table 3.

Table 3: Robustness for zero order and first order method at 266 and 325nm respectively

	Zero order method			First order method	
	Water	**Methanol**		**water**	**Methanol**
SD	0.003215	0.003	**SD**	0.003215	0.003
RSD%	1.909634	1.685393	**RSD%**	1.909634	1.685393

.

4. Sensitivity:

The limit of detection (LOD) and limit of quantification (LOQ) were calculated by using the equations LOD = 3 x σ / S and LOQ = 10 x σ /S, where σ is the standard deviation of intercept, S is the slope. The LOD and LOQ were found to be 0.0898µg/mL and 0.165 µg/m respectively for zero order derivative and The LOD and LOQ were found.

5.Accuracy:

Accuracy of analytical procedure expresses the closeness of agreement between the value which is accepted either as a conventional true value or an accepted reference value and the value found..prepare a 6 concertrations of 32ug/ml, 40ug/ml and 48ug/ml from stock solution of api and pharmceutical formulation.Results obtained are shown in Table 4.

Table 4: Accuracy for zero order and first order method at 266 and 325nm respectively

Actual conc.	Observed concentration	Recovery %	% RSD
Zero Order derivative method			
32	31.5	98.43	0.834
40	40.2	100.5	0.933
48	47.7	99.37	0.615
First Order derivative method			
32	32.4	101.25	0.75
40	39.8	99.5	0.7
48	49.1	102.29	0.383

IV. CONCLUSIONS

A simple, sensitive, precise, accurate, rapid and reproducible UV method has been developed and validated of Silodosine in bulk and in pharmaceutical formulation.

These method is mainly economically because in this method stock solution is prepared by methanol but further dilutions prepared are by Distilled Water.

V. ACKNOWLEDGMENT

The auther thank to JSPM's Charak college of pharmacy and research, wagholi, pune for providing facilities.

REFERENCES

[1] Chaudhari, R. Choksi, V. Divan, T. Patel, B. Parmar, S. 2013. Development and Validation of First Order Derivative Spectrophotometric Method for Estimation of Lacosamide in bulk And Tablet Dosage Form. Asian Journal of Pharmaceutical and Clinical Reserch, 6(3): 162-164.

[2] Khan, F. Gurupadayya, B. Nasefa, A. Prudhvi, S. 2015. Development and Validation of Zero and First Order Spectrophotometric Method for Determination of Opipramol in Bulk and Pharmaceutical Dosage Form Original. International Journal of Pharmacy and Pharmaceutical Sciences, 7(3): 274-277.
[3] Himaja, M. Kalpana, J. Anbarasu, C. 2014. Validated Zero Order and First Order Derivative Spectrophotometric Methods for Invitro Analysis of Tenofovir Disoproxil Fumarate Tablets using Azeotropic Mixture. International Journal of Pharmacy and Pharmaceutical Sciences, 6(6): 302-304.
[4] Ahmed, M. Rajan, M. Shetty, S. Rajesh, M. 2011. Zero order and First order Derivative Spectrophotometric methods for determination of Cisapride in Pharmaceutical formulation. International Journal of ChemTech Research, 3(3):1020-1024.
Adhikari, L. Ghatak, S. Mishra, U. Murthy, P. 2011. Zero order and first order derivative method development and validation of Gliclazide in its bulk and pharmaceutical dosage form. Pelagia Research Library, 2(4): 295-300.
[5] Jahan, K. and Malipatil, M. 2014. Development And Validation Of New Spectrophotometric Methods For The Quantitative Estimation Of Silodosin In Bulk Drug And Pharmaceutical Formulations. International Journal of Pharmaceutical Research & Analysis, 5(4): 65-69.
[6] Aneesh, T. Rajasekaran, A. 2012. Method Development and Validation for the Estimation of Sildosin in Bulk and Pharmaceutical Dosage Forms using Uv-Vis Spectrophotometry. Asian Journal of Pharmaceutical and Clinical Research, 5(4): 150-152
[7] The D. Indian pharmacopoeia. New Pharmacopoeial Commission G International Journal of ChemTech ResearchZhaziabad, 2007, 1276.
[8] International Conference on Harmonization (ICH). 1995. Guidance for Industry: Q2A Validation of analytical procedures. Consensus Guidelines: ICH Hormonized Tripartite Guidelines.
[9] International Conference on Harmonization. 1997. Topic Q2B. Validation of Analytical Methods: Methodology. The Third International Conference on Harmonization of Technical Requirements for Registration of Pharmaceuticals for Human Use (ICH), Yokohama, Japan.
[10] Charwal, G. 2013. Instrumental Methods of Chemical Analysis. Himalaya Publishing House, 5: 2.624-2.631.
Skoog, D. Principles of Instrumental Analysis. Brooks/Cole Pub Co, 6: 728-735.
[11] Willard, E. Instrumental Method of Analysis. CBS Publishers and Distributors, 7: 600.
Jeffery, K. Vogel's Textbook of Quantitative Chemical Analysis. ELBS. Longman Publication England, 5: 224-228.
[12] The Merck Index. 2001. an encyclopedia of chemicals, drugs & biological. Merck research laboratories division of Merck & Co Inc. white house station, 14: 857.
[13] Christian, G. 2013. Analytical Chemistry. Publication of John Wiley and Sons, 6: 1-15.
[14] Sharma, B. 2002. Instrumental Methods of Chemical Analysis. Goel Publication Co., Meerut, 25: 3-6.
[15] Beckett, A. Stenlake, J. 2004. Practical Pharmaceutical Chemistry. Publication of New Delhi, 4: 275-300.