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Sitagliptin and Simvastatin Simultaneous Estimation using RP-HPLC Method

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Abstract: *A simple, specific, accurate, rapid, inexpensive isocratic Reversed Phase-High Performance Liquid Chromatography (RP-HPLC) method was developed and validated for the quantitative determination of Sitagliptin and Simvastatin pharmaceutical tablet dosage forms. RP-HPLC method was developed by using BDS C 18 (150 mm*4.6 mm) column. The mobile phase composed of Buffer: Acetonitrile: Methanol 20:70:10. The flow rate was set to 1.0 mL.min with the responses measured at 215 nm. Retention time of simvastatin and sitagliptin were found to be 4.7 min and 3.1 minutes. Sitagliptin showed linearity between 25 - 150 microgm/ml & Simvastatin showed linearity between 2.5-15 microgm/ml. with correlation coefficient. The validation of the developed method was carried out for specificity, linearity, precision, accuracy, robustness, limit of detection, limit of quantitation. The developed method can be used for routine quality control analysis of in simvastatin and sitagliptin pharmaceutical tablet dosage form.*

Keywords: Simvastatin and Sitagliptin, UV-Vis detector, Method Development & Validation

I. INTRODUCTION

High-performance liquid chromatography also known as **High-pressure** or **High price** or **High speed liquid chromatography**, **HPLC** is a form of column chromatography used frequently in analytical chemistry and biochemistry to identify, separate, and quantify compounds. It is a powerful tool in analysis. It is basically an improved form of column chromatography which has been optimized to provide rapid high resolution separations. Early LC used gravity fed open tubular columns with particles 100s of microns in size; the human eye was used for a detector and separations often took hours or even days to develop.

HPLC requires basic & special modules as follows:

- Solvent reservoir with a Degassing system.
- Extremely precise gradient mixers (optional).
- HPLC high pressure pumps with very constant flow.
- Unique high accuracy, low dispersion, HPLC sample valves.
- Very high efficiency HPLC columns with inert packing materials.
- High sensitivity low dispersion HPLC detectors.
- High speed data acquisition systems.

II. EXPERIMENTAL METHODOLOGY

Table -1: Instrumentation:

1.	Instrument	HPLC
2.	Company	Alliance Waters 2695
3.	Type	Auto sampler

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4.	Type	Column oven &degasser
5.	Detector	2996 PDA detector
6.	Softwere	Empower-2software

Table –2: Reagents and chemicals:

Regents/Chemical	Company
Sitagliptin & Simvastatin	Hetero Pharma
HPLC grade acetonitrile	Merck chemical division
Milli-Qwater	Milli-Q purification system
Tablets	local Pharmacy

Preparation of solutions:

Buffer solution: Accurately measured 1.0 ml of Ortho phosphoric acid in a 1000 ml of volumetric flask, about 900 ml of HPLC grade water obtained from Milli-Q water purification system was added, sonicated and degassed and lastly fabricated the volume to 1000 ml with water.

Standard Stock Solution: Perfectly Weighed & transferred 100mg of Sitagliptin, 50mg of Simvastatin into a 25 ml and 100ml VF-volumetric flask respectively , add diluent to final volume. From this, 1 ml was pipette out to a 10 m1 volumetric f1ask & diluents added to obtain final volume.

Working Standard Solutions: 0.25, 0.5, 0.75, l.0, l.25 & l.5 m1 were pipette out from stock solution & shifted to l0 ml volumetric flask & volume was filled up to l0 ml with diluent. Thisgivessolutionsof25,50,75,100, 125,150 microgm/mlforSitagliptinand2.5, 5.0, 7.5, l0.0, l2.5, l5.0 microgm/ml for Simvastatin respectively.

Sample preparation: 1 tablet was weighed and powdered and it was taken intoa 100 ml volumetric flask – VF and filled with diluents. This was filtered by HPLC filters.1ml of filtered sample stock solution was transferred to 10ml volumetric flask and made up with diluents.

Method Validation: System suitability, Specificity, linearity, precisions, accuracy, robustness, solution stability, LOD, LOQ, assay were evaluated**.**

Table -3: Optimized chromatographic conditions of Sitagliptin & Simvastatin

S.No.	Systems	Values
1	Mobilephasemix	Buffer,Acetonitrile and methanol Taken in the ratio 20:70:l0
2	pH	3.3
3	Column, make	BDS column(4.6xl50mm, 5µm)
4	Column temperature	30°C
5	Wave1ength	215nm
6	Injectionvolume	10µ1
7	Flowrate	l.0m1/min
8	Run time	07 min
9	Retentiontime(Sitagliptin)	3.1 min
10	Retention time(Simvastatin)	4.7 min

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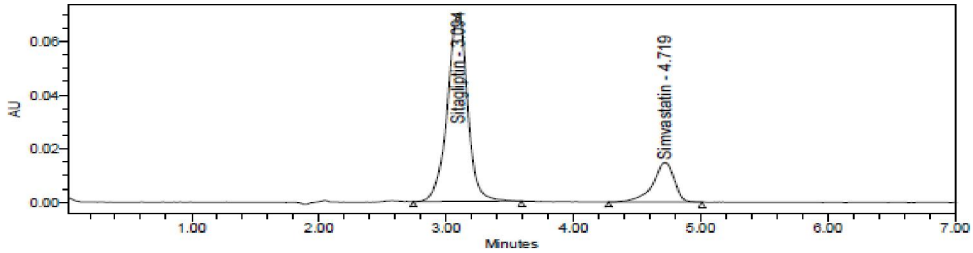

Fig 1: Optimized Chromatogram of Sitagliptin & Simvastatin standards

III. RESULTS AND DISCUSSION

System Suitability: The theoretical plates were > 2000, USP tailing < 2 and USP resolution >2. The % RSD of areas for Sitagliptin & Simvastatin were 0.6 and 0.9 respectively.

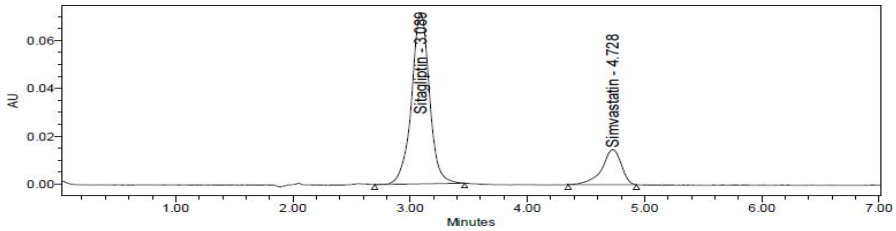

Fig 2: System suitability chromatogram of Sitagliptin & Simvastatin

Specificity: No middle some peak was ascertained at the RT of Sitagliptin & Simvastatin. Thus, the procedure was specific for findout of Sitagliptin & Simvastatin.

Linearity: Sitagliptin showed linearity between 25 - 150 microgm/ml & Simvastatin showed linearity between 2.5- 15 microgm/ml. Linear regression equation was: y (Sitagliptin) = 9038x + 536.2 (r^2=0.999), y (Simvastatin) = 17653x +2482.4 (r^2=0.999) & correlation coefficient (r^2) for Sitagliptin & Simvastatin was >0.99. Hence, this was linear.

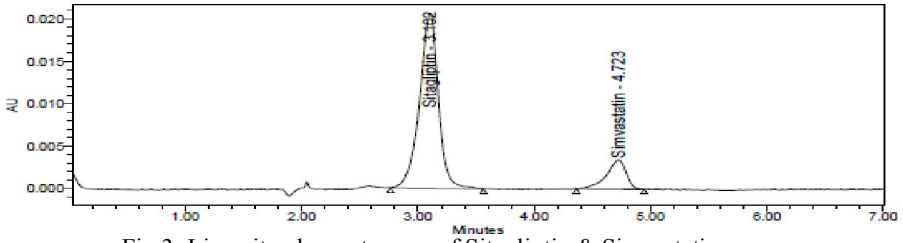

Fig 3: Linearity chromatogram of Sitagliptin & Simvastatin

Table -4: Results of Linearity of Sitagliptin & Simvastatin

Sitagliptin		Simvastatin	
Conc. (microgm/ml)	Peakarea (n=3)	Conc. (microgm/ml)	Peakarea (n=3)
25	226821	2.5	48698
50	456092	5	92342
75	678915	7.5	135218
100	900913	10	176757
125	1122454	12.5	224773
150	1363498	15	266386

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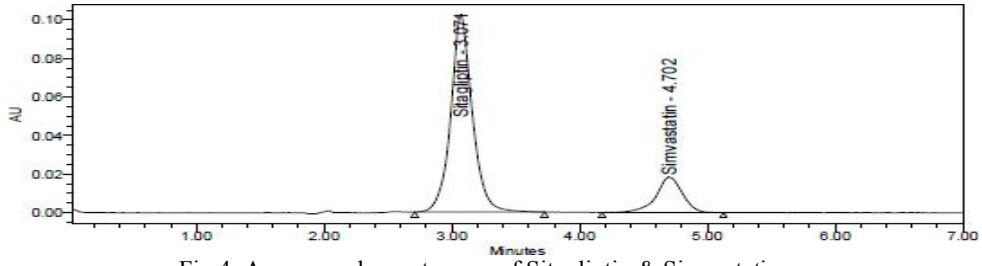

Fig 4: Accuracy chromatogram of Sitagliptin & Simvastatin

Accuracy: Fixed sample solutions added to 50%,100% &150% standard solutions. The %Mean recovery for Sitagliptin & Simvastatin were100.94 &101.02, respectively. %RSD for Sitagliptin & Simvastatin were 0.62 and 1.07. Thus, the method was accurate.

Table -5: Results of Accuracy of Sitagliptin & Simvastatin

Fixedamount (microgm/ml)		SpikedAmount (microgm/ml)		%Recovered	
Sitagliptin	Simvastatin	Sitagliptin	Simvastatin	Sitagliptin	Simvastatin
100	10	50	5	101.07	99.94
100	10	50	5	100.08	102.92
100	10	50	5	101.87	101.40
100	10	100	10	101.43	100.82
100	10	100	10	101.15	100.62
100	10	100	10	100.48	101.12
100	10	150	15	100.78	100.19
100	10	150	15	100.16	99.80
100	10	150	15	101.44	102.41
			MEAN	100.94	101.02
			SD	0.61	1.08
			%RSD	0.62	1.07

Precision: The repeatability and intermediate precision data were summarized in below Table.

Repeatability: The %RSD for Sitagliptin & Simvastatin were 0.6 and1.2. Mean area: 760283 for Sitagliptin, 174419 for simvastatin.

Table - 6: Results of Repeatability of Sitagliptin & Simvastatin

	Sitagliptin			**Simvastatin**		
S.NO	Area	USP Plate Count	USP Tailing	Area	USP Plate Count	USP Tailing
1	754903	2293	1.01	173255	4319	1.00
2	760395	2204	1.00	177112	4416	1.01
3	764412	2259	1.01	172896	4380	1.02
4	756031	2072	1.02	177052	4517	1.02
5	766483	2139	1.00	173772	4534	1.00
6	759481	2179	1.01	172429	4564	1.02
Mean	760283			174419		
Std. Dev.	4542.8			2109		
%RSD	0.6			1.2		

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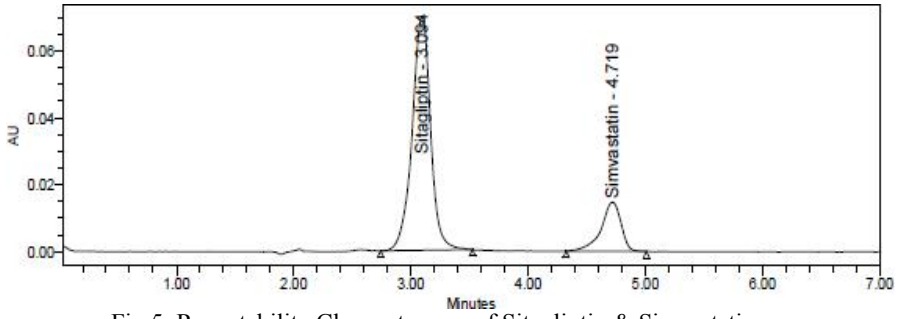

Fig 5: Repeatability Chromatogram of Sitagliptin & Simvastatin

LOD and LOQ: Sitagliptin LOD and LOQ were 0.19 and 0.55 microgm/ml, respectively and for Simvastatin were 0.04 and 0.10microgm/ml.

Table -7: LOD and LOQ data of Sitagliptin & Simvastatin

Sitagliptin				**Simvastatin**		
S.NO	SLOPE	Y-INTERCEPT		S.NO	SLOPE	Y-INTERCEPT
1	8987.1	136.54		1	17666	2456.4
2	9187.6	382.04		2	17590	2663
3	8939.3	1090.1		3	17703	2328.5
AVG	9038	536		AVG	17653	2482
SD		496.5		SD		168.90
LOD		0.19		LOD		0.04
LOQ		0.55		LOQ		0.10

IV. CONCLUSION

A new simple, precise and accurate HPLC method was developed and validated for the simultaneous estimation of Sitagliptin & Simvastatin in marketed formulations. In this method, BDS 150mm x 4.6 mm, 5µ. column was picked as stationary phase. Buffer and acetonitrile,methanol were taken in the ratio 20:70:10%v/v and mobile phase with 1.0 ml/min flow rate. The retention times of Sitagliptin & Simvastatin were found to be 3.1min & 4.7min, respectively. Here, number of theoretical plates were 2285 and 4301, USP tailing were 1.01, 1.02 for Sitagliptin & Simvastatin, respectively; this showed optimized method met the system suitability parameters. Presently, regression coefficient was 0.999 for Sitagliptin & Simvastatin and was linear. The percentage mean recovery of Sitagliptin & Simvastatin were found to be 100.94, and 102.42%, respectively and it showed that the proposed method was accurate. RSD values of Repeatability & intermediate precision were ≤2 and system is precise. The lowest values of LOD and LOQ (0.19 and 0.55µg/ml; 0.04 and 0.10µg/ml for Sitagliptin & Simvastatin, respectively) indicate sensitive. Solutionstability studies of method indicate Sitagliptin & Simvastatin drugs were stable upto 24hours.

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