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RP-HPLC method development and validation for simultaneous estimation of nebivolol and valsartan in pharmaceutical dosage forms

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ABSTRACT

A simple, Accurate, precise method was developed for the simultaneous estimation of the Nebivolol and Valsartan in Tablet dosage form. Chromatogram was run through Std Discovery C18 150 x 4.6 mm, 5□. Mobile phase containing Buffer 0.1%OPA: Acetonitrile taken in the ratio 55:45 was pumped through column at a flow rate of 1 ml/min. Buffer used in this method was 0.1% OPA buffer. Temperature was maintained at 30°C. Optimized wavelength selected was 260.0 nm. Retention time of Nebivolol and Valsartan were found to 2.227 min and 3.126 %RSD of the Nebivolol and Valsartan were and found to be 0.6 and 0.4 respectively. %Recovery was obtained as 99.94% and 100.02% for Nebivolol and Valsartan respectively. LOD, LOQ values obtained from regression equations of Nebivolol and Valsartan were 0.05, 0.16 and 0.18, 0.53 respectively. Regression equation of Nebivolol is y=10542.x+470.4, y = 13049x+16927 of Valsartan. Retention times were decreased and that run time was decreased, so the method developed was simple and economical that can be adopted in regular Quality control test in Industries. Retention times were decreased and run time was decreased, so the method developed was simple and economical that can be adopted in regular Quality control test in Industries.

Keywords: Nebivolol, Valsartan, RP-HPLC

INTRODUCTION

- Specificity
- Linearity
- Range
- Accuracy
- Precision (Repeatability and Ruggedness)
- Detection and Quantitation limit
- Robustness.
- System suitability

The diverse parameters of investigative technique advancement are talked about beneath according to ICH guideline:-

Specificity

Specificity is the capacity to survey unequivocally the analyte within the sight of segments which might be required to be available. Commonly these might incorporate polluting influences, degradants, lattice, and so forth.

Linearity

The linearity of an investigative strategy is its capacity (inside offered extend) to acquire test comes about, which are straightforwardly relative to the focus (sum) of analyte in the example.

Range

The extent of investigative strategy is the interval between the upper and lower obsession (aggregates) of analyte in the case (checking these centers) for which it has been displayed that the logical method has a suitable level of precision, accuracy and linearity. The foreordained range is commonly gotten from linearity ponders and depends upon the arrange dutilization of the philosophy

Accuracy

Precision of explanatory framework conveys the closeness of comprehension between the regard which is recognized either as a conventional honest to goodness regard or a recognized reference regard and the regard found. This is a portion of the time named certainty. Exactness should be developed over the predefined extent of the informative strategy

Precision

The precision of a methodical methodology conveys the closeness of comprehension (level of scatter) between the plan of estimations got from different examining of the same homogeneous case under the prescribed conditions

Detection Limit

The disclosure uttermost ranges of an individual illustrative strategy is the most negligible proportion of analyte in a case, which can be perceived yet not by any stretch of the imagination quantitated under communicated exploratory conditions**7)**

Quantitation Limit

The quantitation uttermost compasses of an individual consistent methodology is portrayed as the most negligible proportion of analyte in an illustration, which can be quantitatively chosen with fitting precision and accuracy.

Robustness

The life of an intelligent system is a proportion of its capacity to remain unaffected by little, yet think about assortments in procedure parameters and gives an indication of its resolute quality in the midst of customary utilize

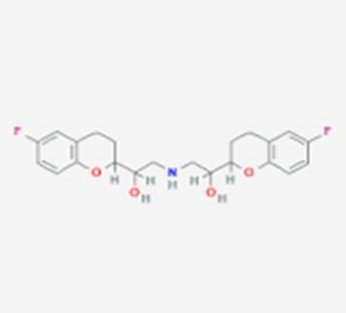
DRUG PROFILE

Nebivolol

Description

Nebivolol is a very cardioselective vasodilatory beta1 receptor blocker utilized in treatment of hypertension. In many nations, this solution is accessible just by medicine. [Wikipedia]

Structure



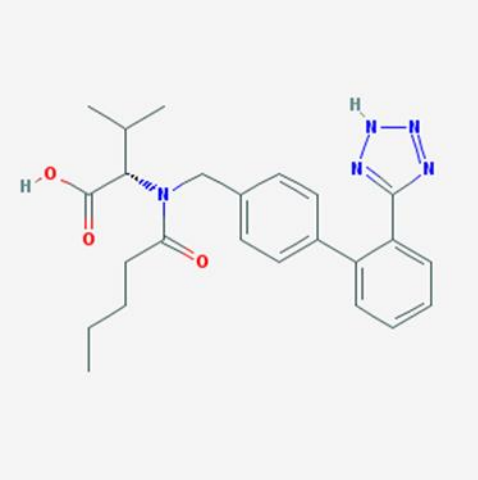
CAS number: 118457-14-0
Weight: Average: 405.435; Monoisotopic: 405.175164703
Chemical Formula: C22H25F2NO4
IUPAC Name: 1-(6-fluoro-3, 4-dihydro-2H-1-benzopyran-2-yl)-2-{[2-(6-fluoro-3,4-dihydro-2H-1-benzopyran-2-yl)-2-hydroxyethyl]amino}ethan-1-ol

Valsartan

Description

Valsartan is an angiotensin-receptor blocker (ARB) that may be used to treat a variety of cardiac conditions including hypertension, diabetic nephropathy and heart failure.

STRUCTURE



CAS number: 137862-53-4
Weight: Average: 435.5188; Monoisotopic: 435.227039819
Chemical Formula: C24H29N5O3
IUPAC Name: (2S)-3-methyl-2-[N-({4-[2-(2H-1,2,3,4-tetrazol-5-yl)phenyl]phenyl}methyl)pentanamido]butanoic acid

MATERIALS AND METHODS

Materials

- Combination Nebivolol and Valsartan tablets (**Nebicard V (5+80)** Torrent Pharmaceuticals Ltd) Received from local pharmacy.
- Refined water, Acetonitrile, Phosphate cushion, Methanol, Potassium dihydrogen ortho phosphate support, Ortho-phosphoric . All the above synthetic concoctions and solvents are from Rankem

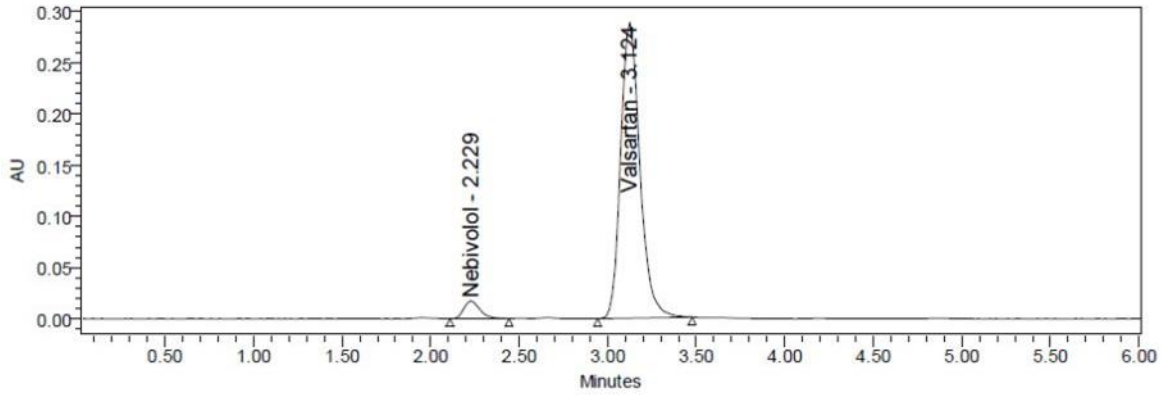
Instruments

- Electronics Balance-Denver
- p^H meter -BVK enterprises, India
- Ultrasonicator-BVK enterprises
- WATERS HPLC 2695 SYSTEM furnished with quaternary pumps, Photo Diode Array finder and Auto sampler coordinated with Empower 2 Software.

RESULTS AND DISCUSSION

Table: 4.1 System suitability parameters for Nebivolol & Valsartan

S no	Nebivolol			Valsartan			
Inj	RT(min)	USP Plate Count	Tailing	RT(min)	USP Plate Count	Tailing	Resolution
1	2.226	2939	1.23	3.119	3927	1.23	4.7
2	2.227	2846	1.25	3.120	4173	1.21	5.0
3	2.227	2927	1.27	3.121	4257	1.24	5.1
4	2.229	2879	1.27	3.124	4121	1.20	5.0
5	2.231	3064	1.26	3.126	3930	1.20	4.9
6	2.232	2982	1.24	3.128	4119	1.23	4.8



DISCUSSION

According to ICH guidelines plate count should be more than 2000, tailing factor should be less than 2 and resolution must be more than 2. All the system suitable parameters were passed and were within the limits.

Table: 4.2 Linearity table for Nebivolol &Valsartan

Nebivolol		Valsartan	
Conc (µg/mL)	Peak area	Conc (µg/mL)	Peak area
0	0	0	0
2.5	26888	40	526830
5	55493	80	1107904
7.5	76971	120	1576529

10	107155	160	2095373
12.5	130640	200	2638564
15	159582	240	3134805

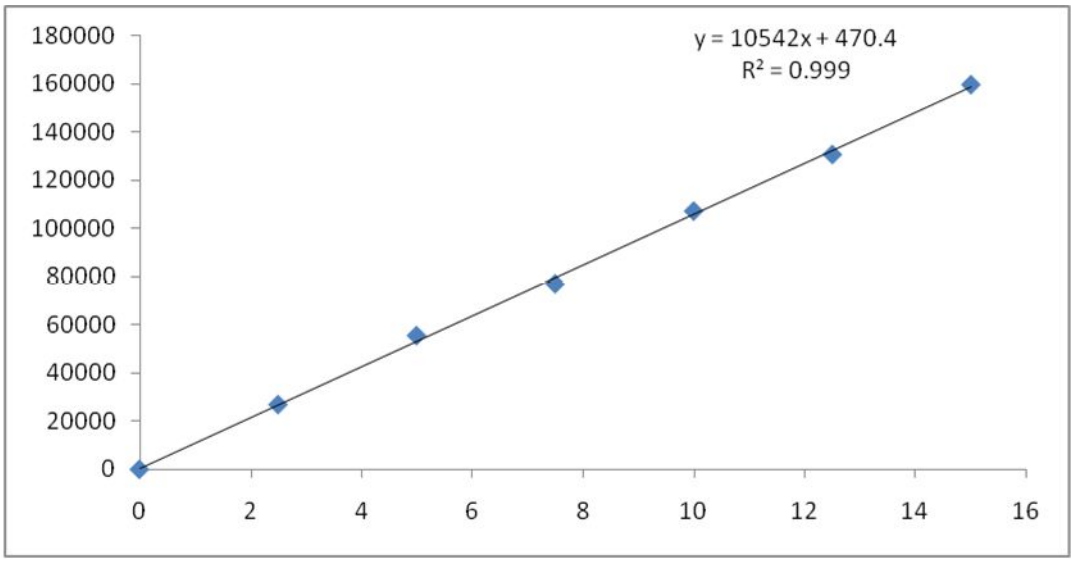


Fig 4.2.1 Calibration curve of Nebivolol

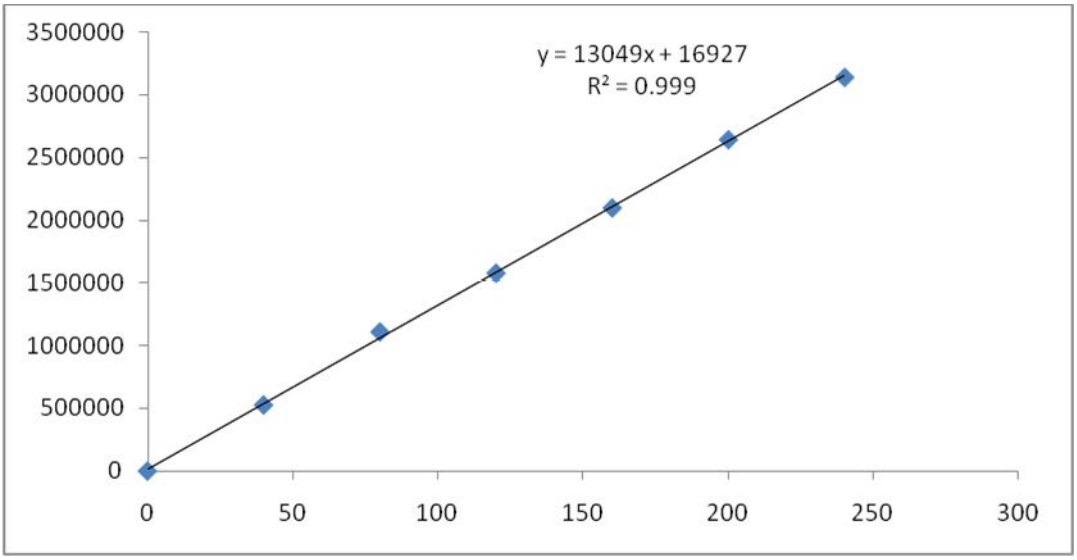


Fig 4.2.2 Calibration curve of Valsartan

DISCUSSION

Six linear concentrations of Nebivolol (2.5-15µg/ml) and Valsartan (40-240µ g/ml) were injected in a duplicate manner. Average areas were mentioned above and linearity equations obtained for Nebivolol was y = 10542.x+ 470.4 and of Valsartan was y = 13049x + 16927 Correlation coefficient obtained was 0.999 for the two drugs.

Table 4.3 Accuracy table of Nebivolol

% Level	Amount Spiked (µg/mL)	Amount recovered (µg/mL)	% Recovery	Mean %Recovery
50%	5	5.07016	101.40	99.94%
	5	4.99266	99.85	
	5	4.98763	99.75	
100%	10	9.92768	99.28	
	10	9.91857	99.19	
	10	9.88955	98.90	
150%	15	15.22563	101.50	
	15	14.98877	99.93	
	15	14.95301	99.69	

SUMMARY AND CONCLUSION

Summary Table

Parameters	Nebivolol	Valsartan	LIMIT
Linearity Range (µg/ml)	2.5-15 µg/ml	40-240 µg/ml	R< 1
Regressioncoefficient	0.999	0.999	
Slope(m)	10542	13049	
Intercept(c)	470.4	16927	
Regression equation (Y=mx+c)	y =10542.x+470.4	y = 13049x+16927	
Assay (% mean assay)	99.33%	99.04%	90-110%
Specificity	Specific	Specific	No interference of any peak

System precision %RSD		0.9	0.4	NMT 2.0%
Method precision %RSD		0.6	0.4	NMT 2.0%
Accuracy %recovery		99.94%	100.02%	98-102%
LOD		0.05	0.18	NMT 3
LOQ		0.16	0.53	NMT 10
Robustness	**FM**	0.2	0.3	%RSD NMT 2.0
	FP	0.6	0.2	
	MM	1.0	0.1	
	MP	0.7	0.5	
	TM	0.7	0.3	
	TP	1.4	0.5	

CONCLUSION

A simple, Accurate, precise method was developed for the simultaneous estimation of the Nebivolol and Valsartan in Tablet dosage form. Retention time of Nebivolol and Valsartan were found to be to 2.227 min and 3.126 %RSD of the Nebivolol and Valsartan were and found to be and 0.4 respectively. %Recovery was obtained as 99.94% and 100.02% for Nebivolol and Valsartan respectively. LOD, LOQ values obtained from regression equations of Nebivolol and Valsartan were 0.05, 0.16 and 0.18, 0.53 respectively. Regression equation of Nebivolol is y=10542.x+470.4, y = 13049x+16927 of Valsartan. Retention times were decreased and that run time was decreased, so the method developed was simple and economical that can be adopted in regular Quality control test in Industries.

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