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Research article

ANALYTICAL METHOD DEVELOPMENT AND VALIDATION BY NEW UPLC METHOD FOR THE DETERMINATION OF SACUBITRIL IN TABLET DOSAGE FORM

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ABSTRACT

A simple accurate, precise rapid isocratic RP-UPLC method development for the simultaneous estimation of Sacubitril in tablet dosage form .The chromatographic system was carried on Sunfire BEH Phenyl(100x2.0 mm) 1.5µm using mobile phase consisting of a 75 volumes of Buffer, 25 volumes of Acetonitrile with detection of 230 nm.. The retention time of Sacubitril was found to be 1.303 min calibration curve was linear over the concentration range of Sacubitril the correlation coefficient for both peak was found to be 0.999 respectively. All the analytical validation parameters were determined and found in the limit as per ICH guidelines.

Keywords: Sacubitril, UPLC.

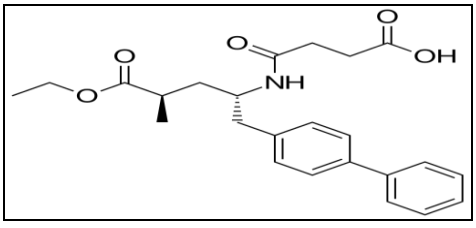
INTRODUCTION

Chromatography is a non-destructive procedure for resolving a multi-component mixture of traces, minor or constituents in to individual fractions. It is a method of separating a mixture of components in to individual components through a porous medium under the influence of solvent.For many years, researchers have looked at "fast LC" as a way to speed up analyses. The need for speed, the availability of affordable and easy to use mass spectrometers. Smaller columns and faster flow rates (amongst other parameters) have been used. Elevated temperature, having the dual advantages of lowering viscosity, and increasing mass transfer by increasing the diffusivity of the analytes, has also been investigated. However, using conventional particle sizes and pressures, limitations are soon reached and compromises must be made, sacrificing resolution. HPLC technology simply doesn't have the capability to take full advantages of sub-2µm particles. UPLC can be regarded as new invention for liquid chromatography.

Review of Literature

N.N.G. Deepika The present work was to develop a fast, accurate, simple, precise, reproducible, Reverse Phase High Performance Liquid Chromatographic method for sacubitril and valsartan by using API Krishna and Shyamalaet. al,

Swathi Vaka A simple and selective LC method is described for the determination of Sacubitril in tablet dosage forms. Chromatographic separation was achieved on a c18 column using mobile phase consisting of a mixture of 80 volumes of methanol and 20 volumes of water with detection of 241 nm.



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Structure for Sacubitril

MATERIALS & METHODS

Table 1. Instrumentation

UV-Visible Spectrophotometer	Nicolet evolution 100
UV-Visible Spectrophotometer software	Vision Pro
UPLC software	Open lab EZ chrome
UPLC	Agilent Technologies
Ultra sonicator	Citizen, Digital Ultrasonic Cleaner
pH meter	Global digital
Electronic balance	Mettler Toledo
Syringe	Hamilton
UPLC Column	Sunfire BEH Phenyl(100x2.0

Table 2. Reagents and Chemicals

Water	HPLC Grade
Methanol	HPLC Grade
Potassium Dihydrogen Phosphate	AR Grade
Acetonitrile	HPLC Grade
Dipotassium hydrogen phosphate	AR Grade
Orthophosphoric acid	HPLC Grade

Working/Reference Standards

Sacubitril Gift samples obtained from Chandra Labs, Hyderabad.

MATERIALS & METHODS

Preparation of Standard Solution of Sacubitril

Accurately weighed about 100mg of Sacubitril and transferred in to100ml of volumetric flask and added 70mL of diluents (Mobile phase used as diluent) and sonicated for 5min and diluted up to the mark with diluent (1000µg/mL)

Then Pipette out 5ml of this solution into 50ml volumetric flask and diluted volume up to the mark with same diluent.

Preparation of Sample Solution of Sacubitril

Sample name: Sacubitril

20 Tablets were weighed and Crushed in motor and pestle and the fine powder of equivalent to 100mg of Sacubitril sample into a 100ml clean volumetric flask added about 70mL of diluents and sonicated up to 20 min for completely dissolved and diluted up to the mark with diluent & mixed well. The prepared solution was filtered through 0.45µ PVDF syringe filter.

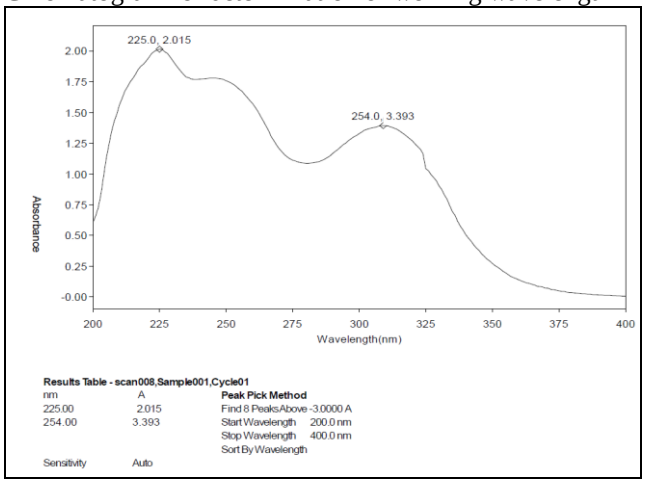
Pipetted 5ml of the above solution into 50ml volumetric flask and diluted volume up to the mark with same diluent.

Chromatographic Conditions

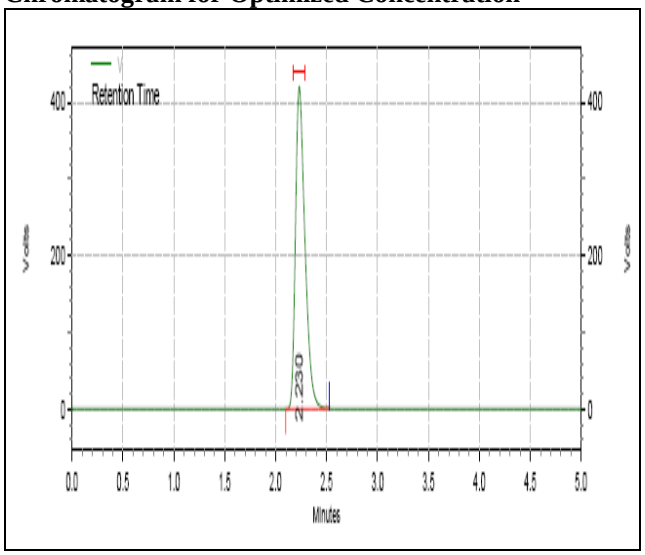
Column	Sunfire BEH Phenyl(100x2.0 mm) 1.5µm
Flow rate	0.5mL /min
Mobile Phase	Ammonium Phosphate Buffer: Methanol(75:25) pH 3.0
Wavelength	230
Injection volume	10µL

RESULT AND DISCUSSION

Chromatogram for determination of working wavelength

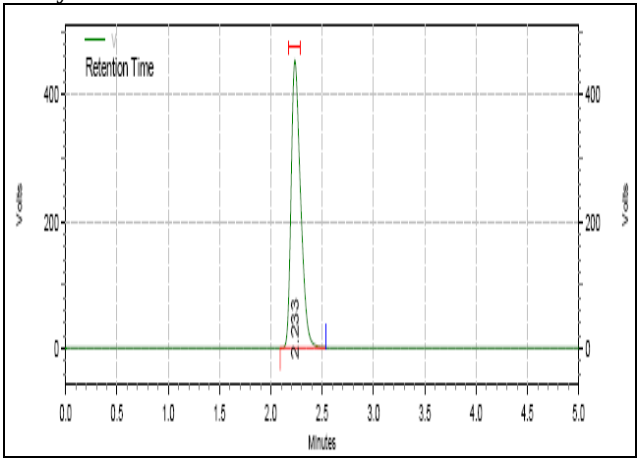


Chromatogram for Optimized Concentration



S. No.	Name	Rt (min)	Peak Area	Theoritical Plates	Tailing Factor	Resolution
1	SACUBI	2.230	45574976	2653	1.4	-

Assay



Chromatogram of Assay Sample Preparation

Table 3. Assay Results

Sacubitril		
	Standard Area	**Sample Area**
Injection-1	45574976	45526367
Injection-2	45376775	45635882
Injection-3	45585138	45728367
Injection-4	45531261	45668237
Injection-5	45554541	45701564
Average Area	45524538.2	45652083.4
Standard deviation	85140.52	
%RSD	0.187	
Assay(%purity)	100.28	

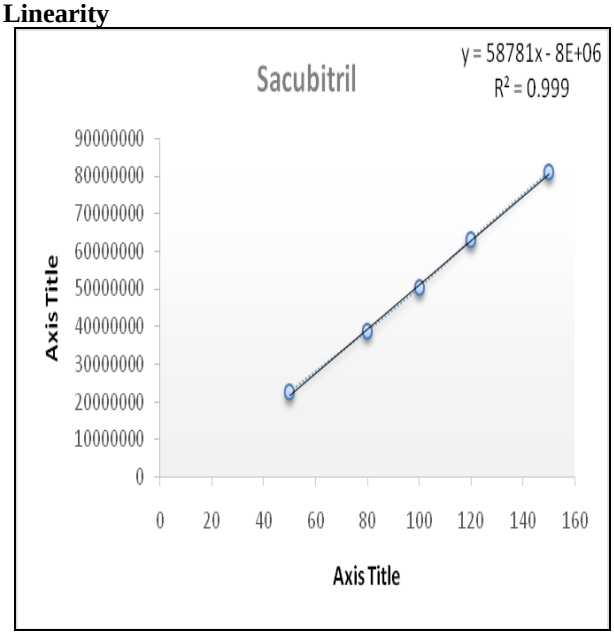
Table 4. Accuracy

Name of the Sample	Standard Weight in mg	Area	Conc Added (µg/ml)	Conc Recovered (µg/ml)	% Recovery	Average
50% Recovery_01	50	23321604	50	50.54	101.1	101.5
50% Recovery_02	50	23336991	50	50.58	101.2	
50% Recovery_03	50	23358809	50	50.62	101.2	
100% Recovery_01	100	47462050	100	102.86	102.9	
100% Recovery_02	100	47460358	100	102.85	102.9	
100% Recovery_03	100	47351113	100	102.62	102.6	
150% Recovery_01	175	81627751	175	176.90	101.1	
150% Recovery_02	175	81192672	175	175.96	100.5	
150% Recovery_03	175	81011385	175	175.56	100.3	

Table 5. Method precision

Injection	SACUBITRIL	
	Area	**%Assay**
1	48190222	102.8
2	48171410	102.6
3	48256935	102.8
4	48146385	102.3
5	48237113	102.3
6	48311273	103.0
Average		102.6
SD		0.30
%RSD		0.3

Linearity



ROBUSTNESS

Table 6. Result of robustness study

Chromatographic changes		Rt(min)	Tailing Factor	Theoretical Plates	%RSD for Standard areas
Flow rate (mL/min)	0.	2.79	1.4	2562	0.3
	0.6	1.867	1.4	2658	0.5
Temperature	2	2.25	1.4	2635	0.9
	3	2.24	1.4	2657	0.9

Table 7. Ruggedness

Intermediate Precision/Ruggedness		
Name of the Standard	**Area**	**%Assay**
Intermediate	48526367	101.2
Intermediate	48635882	101.1
Intermediate	48728367	101.7
Intermediate	48668237	101.1
Intermediate	48701564	101.7
Intermediate	48810447	101.8
	Average	101.4
	Std Deviation	0.35
	%RSD	0.3
% RSD Between %Assay of both Analysts		0.9

DISCUSSION

Assay

The amount of Topiramate present in the taken dosage form was found to be 100.28 % respectively.

Accuracy

The percentage mean recovery of Sacubitril is 101.50% respectively.

System Suitability

The % RSD for the retention times and peak area of Sacubitril were found to be less than 2%.

Linearity and Range

The correlation coefficient for linear curve obtained between concentration vs. Area for standard preparations of Sacubitril is 0.999.

Precision

Test results for Sacubitril are showing that the %RSD of Assay results are within limits.

Robustness

The system suitability parameters were within limit at all variable conditions.

Ruggedness

The %RSD between two analysts Assay values not greater than 2.0%, hence the method was rugged.

CONCLUSION

The validated method is found to be Specific, Linear, Precise, Accurate, Robust and Rugged for the estimation of Sacubitril in tablet dosage form.

Hence it is concluded that the assay method is found to be valid in terms of reliability, precision, accuracy and specificity for routine analysis as well as for stability analysis.

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