



International Journal of Allied Medical Sciences and Clinical Research (IJAMSCR)

ISSN:2347-6567

IJAMSCR |Volume 7 | Issue 1 | Jan - Mar - 2019
www.ijamscr.com

Research article

Medical research

Spectrophotometric method development and validation for the estimation of ticagrelor in bulk and its dosage forms

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ABSTRACT

Simple, sensitive, accurate, precise, stability indicating UV spectrophotometric method have been developed for quantitative determination of ticagrelor in bulk and its dosage form. The UV spectrum was scanned between 200-400 nm and 254 nm was selected as maximum wavelength for absorption. Beer's law was obeyed in the concentration range of 2-10µg/ml were found the method was successfully applied to the pharmaceutical dosage forms containing the above mentioned drug without any interference by excipients. Results of the analysis were validated as per ICH guidelines. This method can be used for the routine and quality control analysis of ticagrelor in raw material and pharmaceutical formulations.

Keywords: Ticagrelor, UV Spectrophotometric method, Validation.

INTRODUCTION

The scope of developing and validating analytical method is to ensure a suitable method for a particular analyte more specific accurate and precise¹. The main objective for that is to improve the conditions and parameters, which should be followed in the development and validation. Ticagrelor is an anticoagulant and anti platelet drug it blocks adenosine diphosphate (ADP) receptors of sub type P2Y₁₂. Ticagrelor is chemically (1S,2S,3R,5S)-3-(7-((1R,2S)-2-(3,4-difluorophenyl)cyclopropylamino)-5-(propylthio)-

3H-(1,2,3)triazolo(4,5-D)pyrimidine-3-yl)-5-(2-hydroxyethoxy)cyclopentane-1,2-diol. Literature review reveals that Ticagrelor lower the risk of thrombotic cardiovascular events in patients suffering from coronary syndrome¹. UV spectrophotometric method is best analytical method for the determination of Ticagrelor¹. Extensive survey revealed that number of methods has been reported for estimation of Ticagrelor². The aim of the present work is to develop a new simple rapid reproducible, inexpensive and efficient linear spectrophotometric method for the estimation of ticagrelor in bulk and its tablet dosage forms.

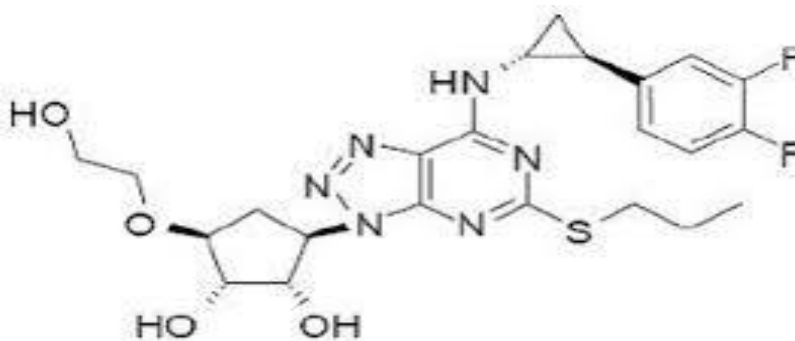


Fig 1: Chemical structure of Ticagrelor

MATERIALS AND METHODS

Ticagrelor dosage form provide AstraZenecapharma India Ltd.(Label claim 90mg) GR Grade acetonitrile and LR Grade Methanol were procured from SD Fine chem Ltd. Water, Whatmann Filter paper.

Apparatus

Weighing balance, UV- Visible Spectrophotometer, (Spectral and absorbance measurements were made on a UV- Visible spectrophotometer with 10mm, Matched pair of quartz cell and spectral band width of ± 2 nm.) Volumetric flask, Pipette, Sonicator, Hot air oven.

UV Method Development

Preparation of Ticagrelor standard stock solution

Stock solution (10 μ g/ml) of Ticagrelor sample was prepared by transferring 10 mg, accurately weighed, into a 10 ml volumetric flask and adding

5 ml acetonitrile .The solution was sonicated for 2 min to dissolve Ticagrelor and the solution was then diluted to volume with the same solvent. Further pipette out 1 ml of this solution and dilute up to 10 ml with acetonitrile .(100 μ g/ml). Further pipette out 2.5 ml of this solution and dilute up to 25 ml with acetonitrile. (10 μ g/ml).

Preparation of Calibration curve

Calibration curve was constructed in accordance with optimum conditions. Aliquot of standard Ticagrelor solution (100 μ g/ml) was transferred into volumetric flask. At respect concentration (2 μ g/ml, 4 μ g/ml, 6 μ g/ml, 8 μ g/ml and 10 μ g/ml) and the absorbance was measured at 254nm.The solution we scanned in the range of 200-400nm against Acetonitrile as blank. A calibration cure was plotted against Absorbance and concentration. The optical characteristics are summarized in Table no 1.

Table no: 1 Calibration curve

S.no	Concentration(μ g/ml)	Absorbance(nm)
1.	2	0.097
2.	4	0.165
3.	6	0.23
4.	8	0.298
5.	10	0.370

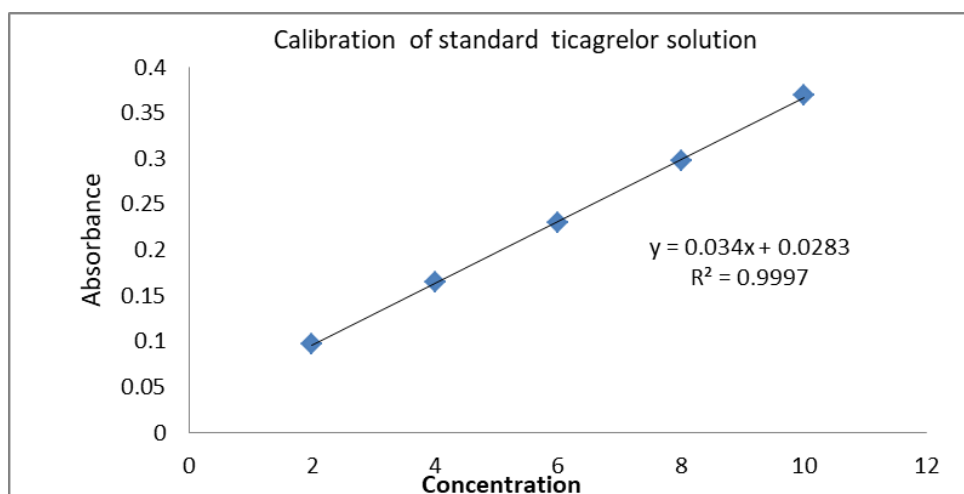


Fig: 2 Determination of λ_{max} of Ticagrelor by UV spectrophotometer

Assay of Ticagrelor in Dosage Forms

Ten tablets were taken and powdered. Weigh a quantity equivalent to 90mg of ticagrelor and transferred into 100ml volumetric flask and dissolve in a small quantity of acetonitrile. The Solution was sonicated and followed by filtration. The first few ml was discarded and the resulting solution is further diluted to produce required concentration. The absorbance was measured at 254nm using Acetonitrile as blank. The maximum

absorbance of sample was found to be 0.21 at 254nm.

UV- method validation

Accuracy

Accuracy should be reported as percent recovery by the assay of known added amount of analyte in the sample or as the difference between the mean and the accepted true value, together with the confidence intervals.

Table no: 2 Accuracy of Ticagrelor

Brand name	Label claim	Amount of drug estimated	%label claim	Standard deviation
Ticagrelor	90mg	89.3	99.2%	.102

Precision

The degree of agreement between an individual test result when the procedure is applied repeatedly to multiple samplings. it is expressed as relative standard deviation.

The reproducibility of the proposed method were determined by performing the tablet assay at

different intervals of same day (intra -day) and three different days (inter day).the result of intra-day and inter-day was expressed in %RSD. Here the study of precision was analysed by measuring the same concentration 6µg/ml in six replicate readings.

Table no: 3 Intraday precision

Concentration(µg/ml)	Absorbance 1	Absorbance 2	Absorbance 3
6µg/ml	0.230	0.234	0.232
6µg/ml	0.232	0.234	0.230
6µg/ml	0.234	0.231	0.230
6µg/ml	0.231	0.230	0.233
6µg/ml	0.230	0.234	0.234
6µg/ml	0.234	0.234	0.232
%RSD	0.78	0.79	0.69
Average% RSD		0.75	

Table no: 4 Inter day precision

Concentration($\mu\text{g/ml}$)	Absorbance 1	Absorbance 2	Absorbance 3
6 $\mu\text{g/ml}$	0.233	0.234	0.232
6 $\mu\text{g/ml}$	0.230	0.232	0.237
6 $\mu\text{g/ml}$	0.234	0.233	0.234
6 $\mu\text{g/ml}$	0.232	0.234	0.235
6 $\mu\text{g/ml}$	0.234	0.235	0.236
6 $\mu\text{g/ml}$	0.232	0.234	0.234
%RSD	0.64	0.76	0.74
Average% RSD	0.71		

Linearity

The linearity of the response of the drug was verified at 2-10 $\mu\text{g/ml}$ concentration. The calibration graph was obtained by plotted the absorbance versus the concentration data and was treated by linear regression analysis..... The

equation of the calibration curve for Ticagrelor obtained $y = 0.034x + 0.028$ the calibration curve was found to be linear in the above mentioned concentrations. The correlation coefficient (r^2) of determination was 0.999.

Table for all parameters

S.no	Parameters	Results
1	Absorption maxima (nm)	254nm
2	Linearity range ($\mu\text{g/ml}$)	2-10
3	Standard regression equation	$y = 0.034x + 0.028$
4	Correlation coefficient (r^2)	0.999
5	Accuracy	102
6	Precision	0.75 (intraday) 0.71 (inter day)

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How to cite this article: P. Sireesha, A. Ajitha, T. Rama Mohan Reddy. Spectrophotometric method development and validation for the estimation of ticagrelor in bulk and its dosage forms. Int J of Allied Med Sci and Clin Res 2019; 7(1): 17-20.

Source of Support: Nil. **Conflict of Interest:** None declared.