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DEVELOPMENT OF NEW VALIDATED METHOD FOR THE DETERMINATION OF SULTAMICILLIN TOSYLATE IN TABLET DOSAGE FORMS BY RP-HPLC

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ABSTRACT

This paper presents a RP-HPLC method for the estimation of Sultamicillin Tosylate in tablets. The process was carried out on C_{18} column (5 µm, 150mm x 4.6 mm, i.d) using phosphate buffer (pH 4.0), acetonitrile in the ratio 60:40 respectively as a mobile phase at a flow rate of 1mL/min. Wavelength was fixed at 230 nm. The retention time of Sultamicillin Tosylate was found to be 9.022. The developed method is rapid, accurate, simple, reliable and sensitive method and it can be used for estimation of the drug in tablets.

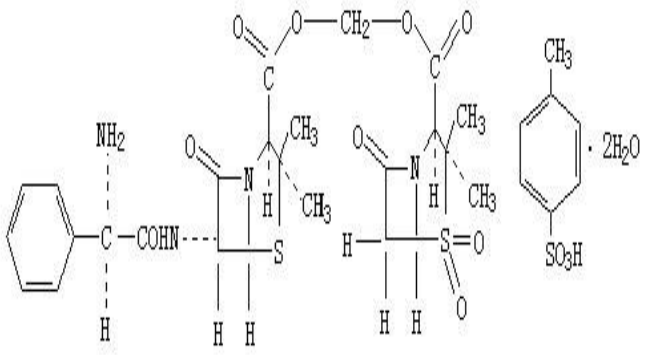
Keywords: Sultamicillin Tosylate RP-HPLC Estimation.

INTRODUCTION

Sultamicillin Tosylate, chemically designated as (2S,5R)- (3,3-dimethyl-4,4,7- trioxo-4-thia-1-azabicyclo [3.2.0] hept2ylcarbonyloxy) methyl (2S,5R,6R)-6-[(R)-2-Amino-2 phenylacetamido] -3,3-dimethyl-7-oxo-4-thia-1-azabicyclo [3.2.0] heptane-2-carboxylate; Hydroxymethyl (+)- (2S, 5R, 6R) -6- [(R)- (2-amino-2phenylacetamido)] -3,3-dimethyl-7-oxo-4-thia-1-azabicyclo[3.2.0] heptane- 2-carboxylate, (2S,5R)- 3,3-dimethyl -7-oxo-4-thia-1-azabicyclo[3.2.0]heptane-2 is a β-lactam antibiotic carboxylate 4,4 dioxide; 4 methyl benzenesulphonic acid; dihydrate. It used against staphylococci that produce beta-lactamase. Sultamicillin has a wide spectrum of activity [1].

From literature survey, it was found that various methods have been reported for the drug but no HPLC methods were reported in any type of pharmaceutical dosage form so far. The present work describes a simple, precise, and accurate reverse phase HPLC method for estimation of Sultamicillin Tosylate in Tablet dosage form. A Validated RP – HPLC Method for Estimation of Sultamicillin Tosylate [2].

Figure 1. Structure of Sultamicillin Tosylate



MATERIALS AND METHODS

Experimental

Phosphate buffer and Acetonitrile used were of HPLC grade and obtained from Merck Chemicals.All other chemicals used were of AR grade and obtained from Sd Fine Chemicals, Mumbai.

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Instrumentation

Quantitative HPLC was performed on a Isocratic HPLC of SHIMADZU prominence consisting of LC – 20AT liquid pump, manual with 20µL sample injection loop and SPD 20A UV-visible absorbance detector. The output – signal was monitored and integrated by Shimadzu spin chrome software. Other different kinds of equipments like analytical weighing balance, ultrasonicator, pH meter, mobile phase reservoir, glassware are used throughout the work.

Chromatographic conditions

The process was carried out on C_{18} column (5µm, 150mm x 4.6 mm, i.d) using the mobile phase consisting of phosphate buffer (pH 4.0), phosphate buffer and acetonitrile, in ratio (60:40 v/v) respectively at a flow rate of 1mL/minutes. Wavelength was fixed at 230 nm. The mobile phase was filtered through 0.2 µ membrane filter and degassed [3].

Preparation of standard solutions

Standard solution of the pure drug was prepared by dissolving 100 mg of Sultamicillin Tosylate in a 100 mL volumetric flask using 25 mL of methanol. Then, the volume made up to the mark with the same solvent. Appropriate volume from this solution was further diluted to get appropriate concentration levels according to the requirement [4].

Preparation of Sample solution

Twenty tablets were weighed the average weight was determined and these were powdered. Sample solution was then prepared by dissolving the powdered tablets equivalent to 100 mg of Sultamicillin Tosylate in a 100 mL of volumetric flask. Then, the drugs were dissolved by using 25 mL acetonitrile and buffer and the volume was made up to the mark. 5 mL of this solution was further diluted to 25 mL with the same solvent. 20µL of solution was injected into HPLC system to obtain chromatogram for standard drug solution and sample solution [5].

Assay method

With the optimized chromatographic conditions, a steady baseline was recorded, the standard solution was injected and the chromatogram was recorded. The retention time of Sultamicillin Tosylate was found to be 9.022 min respectively. This procedure was repeated for the sample solution obtained from the recovery studies.

Method validation

Linearity

Linearity and range of method was determined on standard solution by analyzing 80 to 120 % of test concentration, and the calibration curve was plotted using AUC versus concentration of standard solution.

Accuracy

Accuracy of method was ascertained by recovery study by adding a known amount of standard drug to pre-analyzed sample and reanalyzing the samples by the proposed method.

Precision

Precision was studied by analyzing five replicates of standard solution.

Robustness

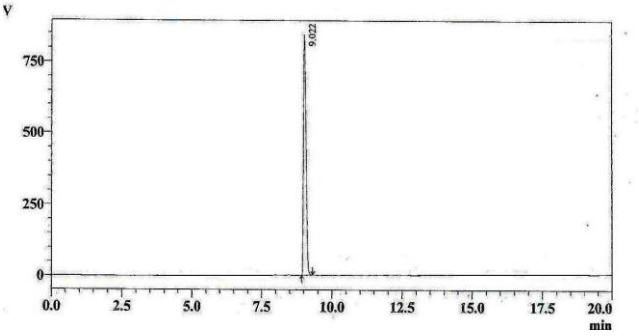
Robustness of method was evaluated by performing the assay with variations in wavelength, pH and flow rate. The chromatographic parameters were also validated by system suitability studies which were carried out on freshly prepared standard stock solution.

RESULTS AND DISCUSSION

The typical chromatogram obtained from the formulation is presented in Figure 1. The retention time for was found to be 9.022 minutes respectively. Asymmetry factor less than 2.00. Linearity was observed in the concentration range of 160 -240 µg/mL, with the correlation coefficient of 0.999 Sultamicillin Tosylate. Accuracy of the method was ascertained by recovery study (n=3). The concentration of standard spiked to the sample was 80% - 120% of the assay level. The method was found to be accurate with percent recoveries between 99.99% and 102.24%. There was good repeatability of proposed method with percentage RSD 0.69 for Sultamicillin Tosylate.

Parameter	Sultamicillin Tosylate
Calibration range	160-240
Theoretical plates	6254
Resolution	8.25
Tailing factor	0.53
% RSD	0.69

Figure 2. Typical Chromatogram of Sample Solution



CONCLUSION

The proposed method was found to be simple, precise, accurate and rapid for determination of

Sultamicillin Tosylate from tablets. The mobile phase is simple to prepare and economical. The sample recoveries in all formulations were in good agreement with their respective label claims. Hence, it can be easily and conveniently adopted for routine analysis of sultamicillin Tosylate in tablets.

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