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REVERSE PHASE HPLC METHOD FOR THE DETERMINATION OF CEFIXIME IN PHARMACEUTICAL DOSAGE FORMS.

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ABSTRACT

A simple, rapid and reproducible high performance reverse phase liquid chromatography method has been developed for the estimation of cefixime in bulk drug sample and pharmaceutical dosage forms was developed using SS aokosil II C18, 250X4.6mm, 5µm column with mobile phase composition of acetonitrile methanol and 0.5% ammonium acetate 44:16:40 (PH 5). Flow rate of 0.8ml/min and uv detection at 295nm linearity was observed over concentration range of 20-100µg/ml. the accuracy of the proposed method was determined by recovery studies and found to be 95-105% . The proposed method was validated and results conformed to ICH parameters.

Key words: cefixime, RP-HPLC

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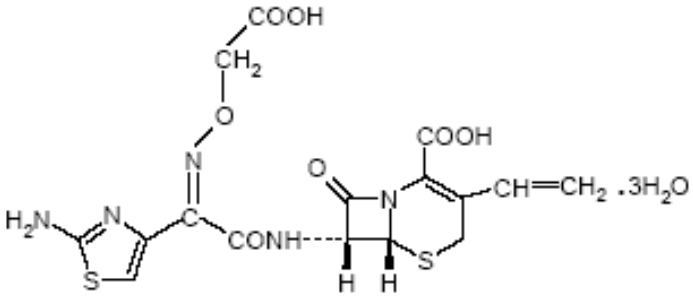
INTRODUCTION



Cefixime is chemically

(6*R*,7*R*)-7-[2-(2-Amino-4-thiazolyl)glyoxylamido]-8-oxo-3-vinyl-5-thia-1-azabicyclo[4.2.0]oct-2-ene-2-carboxylic acid

Which is used to treat many different types of bacterial infections such as bronchitis, tonsillitis ear infections, skin infections, gonorrhea, and urinary tract infections. It is indicated for the treatment of gonorrhea and uninary tract infections. A tablet formulation containing 100mg of cefixime is available (ceftas division of Intas ltd). A survey revealed that no official method is available for the estimation of cefixime by HPLC method. Present work describes the development of a simple, precise and accurate reverse phase HPLC method for estimation of cefixime in tablets.

The drug sample of cefixime were obtained as a gift sample from Intas

EXPERIMENTAL

Material and methods:

Instrumentation

A isocratic high pressure liquid chromatography shimadzu 10AT, SPD 10A detector was used for study .the column used was reverse phase. SS wakosil II, C18, 250X4.6mm, 5mm i.d & partical size 5µm. The flow rate of mobile phase was maintained at 0.8ml/min and detection was carried out at 220nm at the room temp.

Chemical and regents

Water of HPLC grade was collected from milli-Q system acetonitrile (Ranbaxy) methanol(ranbaxy) and ammonium acetate AR (Ranbaxy).

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Preparation of mobile phase

440ml of acetonitrile, 160ml of methanol was mixed with 400ml of ammonium acetate buffer the pH was adjusted to 5.54 with acetic acid, filtered & degassed through nylon membrane filter paper and sonicated for 10min.

Preparation of standard solution

Standard solution of cefixime was prepared in mobile phase of concentration 500µg/ml. the stock solution were diluted to obtain working standard solution of concentration of 20 µg/ml to 100 µg/ml. the resulting solution were sonicated for 10min . 100 µl of the standard solution was injected the retention time for cefixime was found to be 3.17min. The linearity ranges for cefixime was found to 20 - 100 µg/ml.

Preparation of sample solution

Ceftas, tablets five in number were weighed an amount equivalent of 5mg of cefixime was transferred into 10ml volumetric flask. The powder as first dissolved with a few drops of mobile phase and the volume then made upto10ml with mobile phase. The solution was filtered through membrane filter with pore size of 0.45 micron. The sample stock solution was adequately dilute to obtain cefixime concentration of 10 µg/ml. The resulting solution was sonicated for 10min and 100 µl of the sample was injected. The peak area from the chromatogram was tabulated and amount of cefixime present in the tablet formulation was determined from the linearity curve.

Table-1

Drug	Amount added	Amount recovered	Average recovery%
Cefixime	20	9.5	95
	40	15.9	103
	60	21.0	105

Table -2

Parameter	Cefixime
Theoretical plates	8864 per meter,2216 per column for cefixime
Tailing factor	0.6
Resolution	0.632
Calibration range	20 – 100 µg/ml

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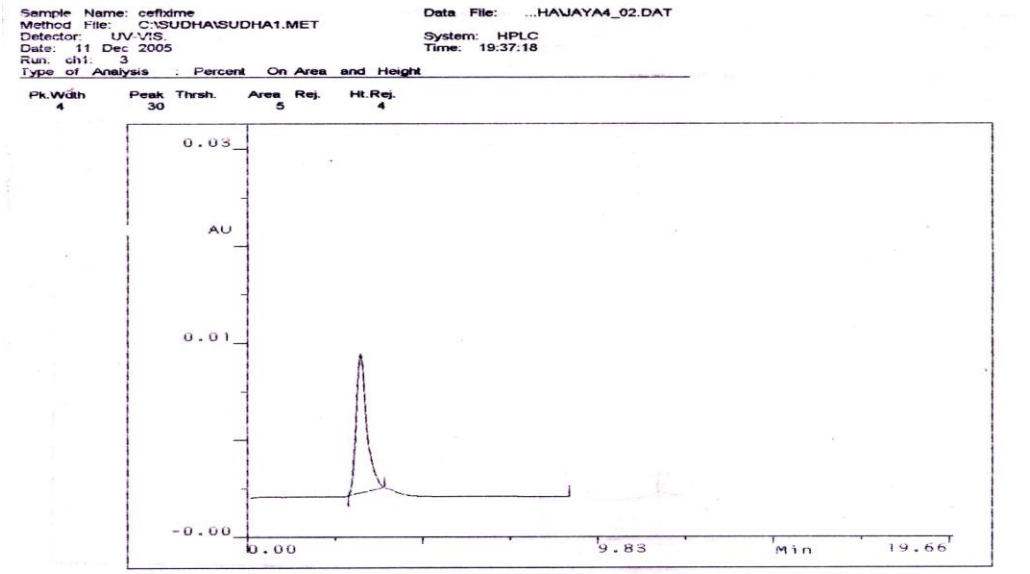
Chromatogram of sample solution



No.	Retn. Time	Height	Area	Height %	Area %	Peak Type
1	8.17	2547	1336479	100.0000	100.0000	BB
		3e+03	1336479			

SUMMARY

TOTAL PEAKS	1
MUL. FACTOR	1.0000
SAMPLE AMT.	100.0000
DILUTION	1.0000

RESULTS AND DISCUSSION

The proposed method was validated as per ICH parameter precision of the proposed HPLC method was carried out by injecting replicate of six of concentration 10 µg/ml and the precision of the proposed HPLC method was found to be 0.79% for cefixime. The RSD values indicate that the proposed method had good precision. Accuracy of the method was also determined. The average recovery of cefixime was 98.6-99.8% respectively. The sample recovery in the formulation was in good agreement with the label claim. High percentage recovery showed that the method was free from interferences of the excipients used in the formulations ruggedness of the method was determined by carrying out the assay by different analysts on different days. The test results were found to be satisfactory with RSD for set of analysis on the same date being less than 0.48%. The percentage area on calculation was found to be 99 to 101% for cefixime. This shows that the results are reproducible. Robustness of the method was determined by carrying out the assayduring which the mobile phase ratio and pH of mobile phase were altered slightly. The percentage recovery found to be 98 – 99% for cefixime is given in the table -2. Assay of the cefixime in tablet dosage form was found to 99.8% of cefixime [1-6].

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CONCULUSION

The method was simple and had short run time of 3.17min, which makes the method rapid . the results of the study indicate that the proposed HPLC method was simple, precise, highly accurate, specific and less time consuming

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