RESEARCH PAPER | Science | Volume : 5 | Issue : 5 | May 2015 | ISSN - 2249-555X

Charectarization and Validation of Cefixime Trihydrate Tablets with Ftir and Rp-Hplc Techeniques

KEYWORDS: Cefixime Trihydrate, HPLC, IR

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ABSTRACT *Drug compatibility with excipients was checked by HPLC and FTIR studies. An accurate, Precise, Simple and Economical High Performance Liquid Chromatographic method for the estimation of Cefixime was developed and validated. The method so developed is Reverse Phase High Performance Liquid Chromatographic method using shimadzu (Japan) C-18 (5µ, 250×4.6mm) with a tetrabutyal ammonium hydroxide buffer adjusted with Ortho phosphoric acid (pH 6.5). Flow rate of 1ml/min and UV detection at 254nm linearity was observed over concentration range of 100-300 µg/ml. the accuracy of the proposed method was determined by recovery studies and found to be 95-101%. The proposed method was validated and results conformed to ICH parameters.*

Introduction

Cefixime (CEF) is an oral third generation cephalosporin antibiotic (Wilson et al 1998 and Suchetha Raddy et al 2011). Chemically, it is (*6R,7R*)-7-{[2-(2-amino-1,3-thiazol-4-yl)-2-(carboxymethoxyimino)acetyl]amino}-3-ethenyl-8-oxo-5-thia-1-azabicyclo-[4.2.0]oct-2-ene-2-carboxylic acid (Sudhakar et al 2010 and N.G. Raghavendra Rao et al 2011), is an orally absorbed third generation cephalosporin antibiotic. It has a broad antibacterial spectrum against various gram-positive bacteria and gram-negative bacteria(Elsadig et al 2012 and Mahesh M. Deshpande et al 2010), clinically used in the treatment of susceptible infections including gonorrhea, otitis media, pharyngitis, lower respiratory-tract infections such as bronchitis, and urinary-tract infections (Mc Millan et al 2007 and Adam D et al 1995 and S.C. Arora et al 2010).

Material and method-

Ciprofloxacin hydrochloride (standard) were purchased from IFPRESS, Mumbai (India). Acetonitrile and Tetrabeutyal ammonium hydraoxide (HPLC grade). Triethylamine and Orthophasphoric acid (HPLC and analytical grade) , KBr (Potassium bromide) were purchased from E.mark and Quilizan. Water was deionised and triple distilled.

FTIR Spectroscopic Analysis

The FTIR imaging in the present investigation was carried out using a (Bruker, Alpha). KBr pellet method was used for sample preparation for FTIR study. All samples were subjected to FTIR spectroscopic studies to determine the functional groups of the samples. The scanning range was 400-4000 cm-1 for and the resolution was 4cm-1(T.Satyanarayana et al 2012).

HPLC Analysis

Validation of Ciprofloxacin Hydrochloride and Cefixime Trihydrate : The method was validated for the parameters like system suitability, linearity, accuracy, precision, LOD and LOQ.

Instrumentation and Chromatographic Condition: The instrument Shimadzu (Japan), equipped with an LC 2010 HPLC system, Visible detector and pump are inbuilt in LC 2010 HPLC system a auto sampler CHPT 2010 HPLC fitted with 10µl volume sample loop.

Chromatographic Condition for Cefixime Trihydrate: The separation was achieved from C18 column (5µ,250x4.6mm) at 300C temperature with a mobile phase consisting of 30 volume of Tetrabutyal ammonium Hydroxide solution prepared by diluting 25ml of 0.4m (TBAH) solution to 1000ml with water, pH adjusted for orthophosphoric acid (pH 6.5±0.1) and 10 volume of acetonitrile. The mobile phase was filtered through nylon 0.45µm-77mm membrane filter. The Flow rate was maintained 1ml/min. The column effluent was monitored on UV detector set as 254nm and the summarized data were showed in table-5.

Procedure of Cefixime Trihydrate (Standard): A stock solution of cefixime trihydrate (100µg/ml) was prepared by dissolving 10 mg cefixime trihydrate in 100ml mobile phase. Several aliquots of standard solution of cefixime trihydrate was taken in different 100ml volumetric flask and diluted up to the mark with mobile phase to get five different concentration (100, 150, 200, 250 and 300 µgm/ml).

Preparation of Sample : 10 tablets of selected marketed brand of cefixime trihydrate were weighted separately. Their average weight was calculated. Powder of tablets equivalent to100mg of cefixime trihydrate was weighed and taken in a 100 ml volumetric flask and dissolved in 10ml mobile phase, sonicated for about 2-3 min and then filtered for nylon (0.22) µm filter paper. The filtered solution was further diluted in the mobile phase to make the final concentration of working sample equivalent to 100% of target concentration.

Accuracy: According to IP standard of cefixime trihydrate tablets of contain equivalent to not less 95.0 % and not more than 101.0 %.

Result and discussion-

FTIR of Cefixime Trihydrate (Standard) : IR spectrum of CFX is characterized by principal absorption peaks at

Volume : 5 | Issue : 5 | May 2015 | ISSN - 2249-555X

3284 cm^{-1} (-NH_2 primary amine) ,1667 cm^{-1} (N-H stretch), 1540 cm−1 (C-H), , 1669 cm^{-1} (C=O stretching amide), 1591 cm^{-1} (C=N stretching), 1382 cm^{-1} (N-O stretching) (D. S. Saindane et al 2010). Results were show in table-1 and graphical represent were shown in fig-1

FTIR of Cefixime Trihydrate (Samples) : The FTIR spectrum of cefixime trihydrate exhibited characteristic absorption peaks at 3284 cm-1 (-NH2 stretch), 1771-1770 cm-1 (-C=O carboxylic acid), 1670-1669cm-1 (stretching of amide, carbamate), 1541-1540 cm-1 (aliphatic C-H stretching), 1594-1592 cm-1 (oxime C=N stretching) and 1383-1381 cm-1 (N-O stretching). Results are show table-1 and fig-2.

HPLC Analysis of Cefixime Trihydrate :

System Suitability: Chromatographic data are that the retention time for cefixime trihydrate (8.3min, SD 0.32 with % RSD 0.3866), and peak area (mean 9172251.8, SD 3625464 with % RSD 0.395) and theoretical plate number is (mean 6443, SD 63.59,with % RSD .0175) are found. Result were shown table-2 and fig-3,4.

Precision: For all five concentration levels % RSD obtained is less then 1.0%.

Linearity: The linear regration of cefixime trihydrate was found to be Y= 45861. The correlation coefficient value were found to be R2 = 0.9091 for cefixime trihydrate (fig-5).

LOD and LOQ : LOD value for cefixime trihydrate were found to be 199 µg/ml and LOQ value for cefixime trihydrate were found to be 604 µg/ml the results were show in table-1.

Conclusion –

HPLC and FTIR result indicate that all the tablets included in this study seems to have very good bioavailability, all of them comply with Indian pharmacopeia. FTIR spectrum of all the studied medicines shows same properties such as standard formulation of medicine. It is concluded that are all drug supplied in the studies area is fit for the human intake as well as other use.

Table: FTIR Studies of Cefixime Trihydrate (Standard)

Peak (cm-1)	Group	Peak assignment	Sample
3284	-NH2	Primary amine	3284
1771	-C=O	Non conjugated carboxylic acid where as the second band which is expects to shift to lower frequency owing the conjugation appears as a overlapping.	1770
1667	N-H	Acyclic amide	1662
1540	C-H	Stretching	1540
1669	C=O	Stretching amide	1670
1591	C=N	Stretching (oxime)	1592
1382	N-O	Stretching	1383

Table 2: Result of Validation Parameters of Cefixime Trihydrate

Parameter	Result
SD	3625464
Linearity (R^2)	0.09091
Y-intercept	45861
Slop of segregation line	45858.9
% RSD (Indicates precision)	0.7904%
Limit of detection	1994005
Limit of quantification	6042444
Rang	100-300µgm/ml

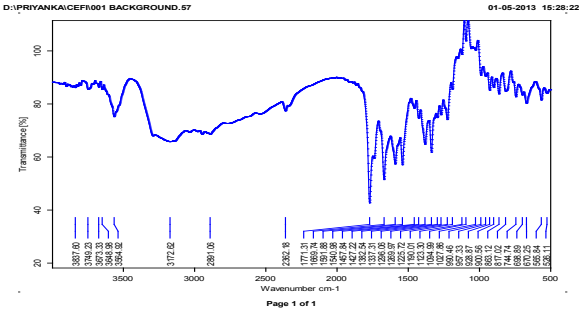

Fig. 1 : FTIR Spectrum of Cefixime Trihydrate (Standard).

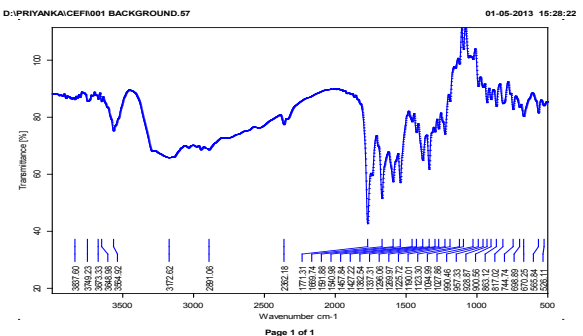

Fig. 2: FTIR Spectrum of sample.

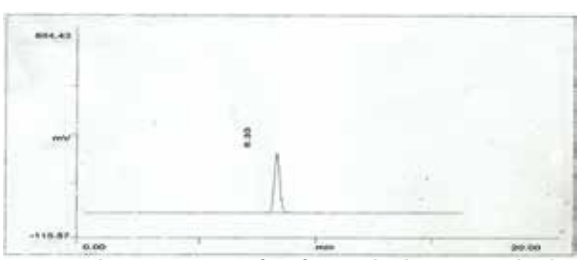

Fig. 3 : Chromotogram of Cefixie Trihydrate (Standard).

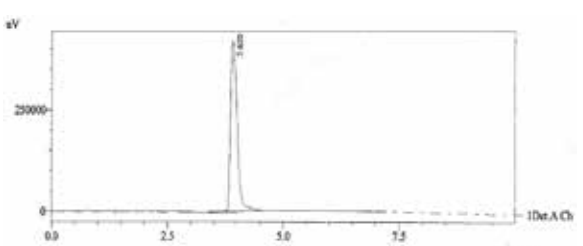

Fig. 4: Chromotogram of Cefixime Trihydrate(Sample).

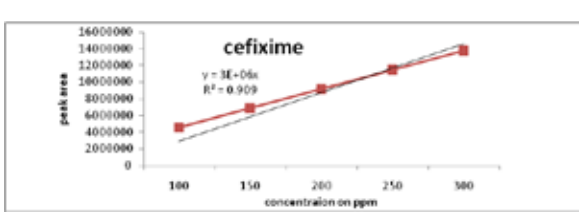

Fig.5: Calibration Curve For Regressed Peak area Value Versus Cefixime Trihydrate Concentration

Volume : 5 | Issue : 5 | May 2015 | ISSN - 2249-555X

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