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ANALYTICAL METHOD DEVELOPMENT AND VALIDATION OF CEFIXIME AND DICLOXACILLIN TABLETS BY RP-HPLC

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ABSTRACT

A rapid and sensitive RP- HPLC[1,2] method with UV-150 detection (220nm) for routine analysis of Cefixime and Dicloxacillin tablet formulation was developed, chromatography was performed with mobile phase containing a mixture of potassium hydroxide buffer and acetonitrile (60:40 v/v) using column C18 – inertstil with flow rate 1.0ml/min in the range of 20ml. In the linearity range of 60-140 µg/ml, cefixime and Dicloxacillin shows a correlation coefficient of 0.9959 and 0.9949 respectively. The proposed method was validated as per ICH Guidelines.[3, 4]

Keywords: Cefixime, Dicloxacillin, RP-HPLC, Validation.

INTRODUCTION

Cefixime[5,6,]7-[2-(2-Aminothiazol-4-yl)2(carboxymethoxyamino)acetoamido]3vinyl--3cephem-4-Carboxlic acid trihydrate as a third generation cephalosporin anti bacterial drug given by oral route in the treatment of susceptible infections including gonorrhea, otitis media, pharynsitis and urinary tract infection. On the other hand administration of cefixime may result in various adverse effects of GIT disturbances and diarrhea. Dicloxacillin[5,6](6R)-6-[-3-(2,6-dichlorophenyl)-5-methylisoxazole-4-Carboxamido] pencillanic acid is Used primarily for the treatment of infections due to *Staphylococci* resistant to benzyl penicillin. This includes bone and joint infections, endocarditic, pneumonia, skin infections and toxic shock syndrome. Dicloxacillin may result in various adverse reactions like hepatitis and cholestatic jaundice have been reported. According to literature survey [7-11] cefixime and dicloxacillin tablet as a combination of active principle in various pharmaceuticals forms were done but pharmacopeias have not yet provided an official method for its quantification. Since there is no systematic method reported for cefixime and diocloxacillin tablets, a simple sensitive and precise method was developed and validated for cefixime and diocloxacillin tablets by RP-HPLC"using UV 150 detector .The developed methods were partially validated as per ICH guidelines.

EXPERIMENTAL

Quantitative HPLC was performed on a isocratic high pressure liquid chromatography (HPLC spectra series p100) with single pan balance (shimadzu libror AEG-220), with pH meter HP-1- PLUS (SESIM) detector UV 150 and reverse phase C-18 column (250x 4.5mm) inertsil5µ was used. The HPLC system was equipped with chromatographic data software – Iris 32

Reagents and Chemicals

Water (HPLC grade), acetonitrile (HPLC grade), potassium di-hydrogen orthophosphate –RANKEM and potassium hydroxide –RANKEM

PARAMETERS	CONDITIONS
Instrument	Spectra Series P 100
Column	C 18-Inertsil
Wavelength	220 nm
Temperature	Room Tempe
Flow Rate	1.0ml/Min
Injection Volume	20µl
Mobile Phase	Buffer: ACN (60:40)
Run Time	10 Min
Detector	UV150

Chromatographic conditions

Preparation of standard solution of cefixime:

55mg of cefixime WS was weighed and transferred to 100 ml volumetric flask, add 40 ml of mobile phase make completely dissolve by sonic ate. Make up the volume with mobile phase.

Preparation of standard solution of Dicloxacillin:

125mg of Dicloxacillin WS was weighed and transferred to 100 ml volumetric flask and add 40ml of mobile phase. Pipette out 5 ml into a 50 ml volumetric flask and make up the volume with mobile phase.

Preparation of test solution of cefixime:

304.25mg of cefixime was weighed and transferred to 100ml volumetric flask and add 40 ml of mobile phase make completely dissolve by sonic ate. Make up the volume with mobile phase. Pipette out 5ml in to 50 ml volumetric flask and make up the volume with mobile phase.

Preparation of test solution of Dicloxacillin:

276.75mg of Dicloxacillin was weighed and transferred to 100ml volumetric flask and add 40 ml of mobile phase make completely dissolve by sonic ate . Make up the volume with mobile phase. Pipette out 5 ml into 50ml volumetric flask and make up the volume with mobile phase.

RESULTS AND DISCUSSION

PARAMETERS	ACCEPTANCE CRITERIA	CEFIXIME	DICLOXACILLIN
Accuracy	% recovery should be between 98.0 to 102.0	% recovery was found to be 99.0 to100.0	% recovery was found to be 99.0 to100
Precision	RSD should not be more than 2%	% of RSD was found to be the 0.379	The % of RSD was found to be the 0.46
Linearity	Correlation coefficient should be not less than 0.99	Correlation coefficient was found to be0.9959	Correlation coefficient was found to be 0.9949
	% curve fitting should not less than 99.90	% curve fitting was found to 99.95	% curve fitting was found to 99.94
Specificity	The tailing factor should not be less than 2.0	The tailing factor was found to be1.1	The tailing factor was found to be 1.1%
	The R_1 determined for cefixime is 2.23 and Dicloxacillin is 6.89	The R_1 determined for cefixime is 2.232	The R_1 determined for Dicloxacillin is 6.891

Ruggedness	% of RSD of assay result obtained should be NMT 2.o	The results were found to be will within the limit	The results were found to be will within the limit
Robustness	The tailing factor should not be less than 2.0	The tailing factor was found to be 1.1%	The tailing factor was found to be 1.1%

ACKNOWLEDGMENTS

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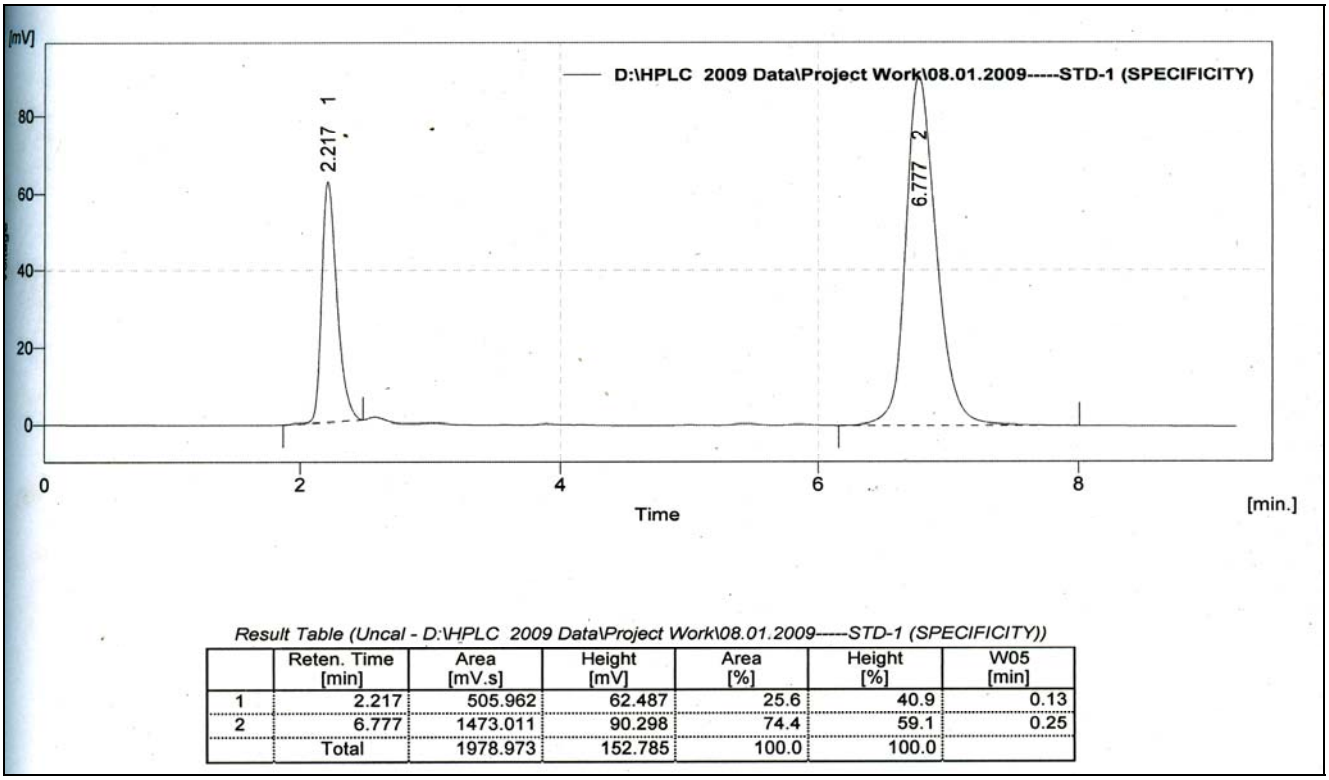
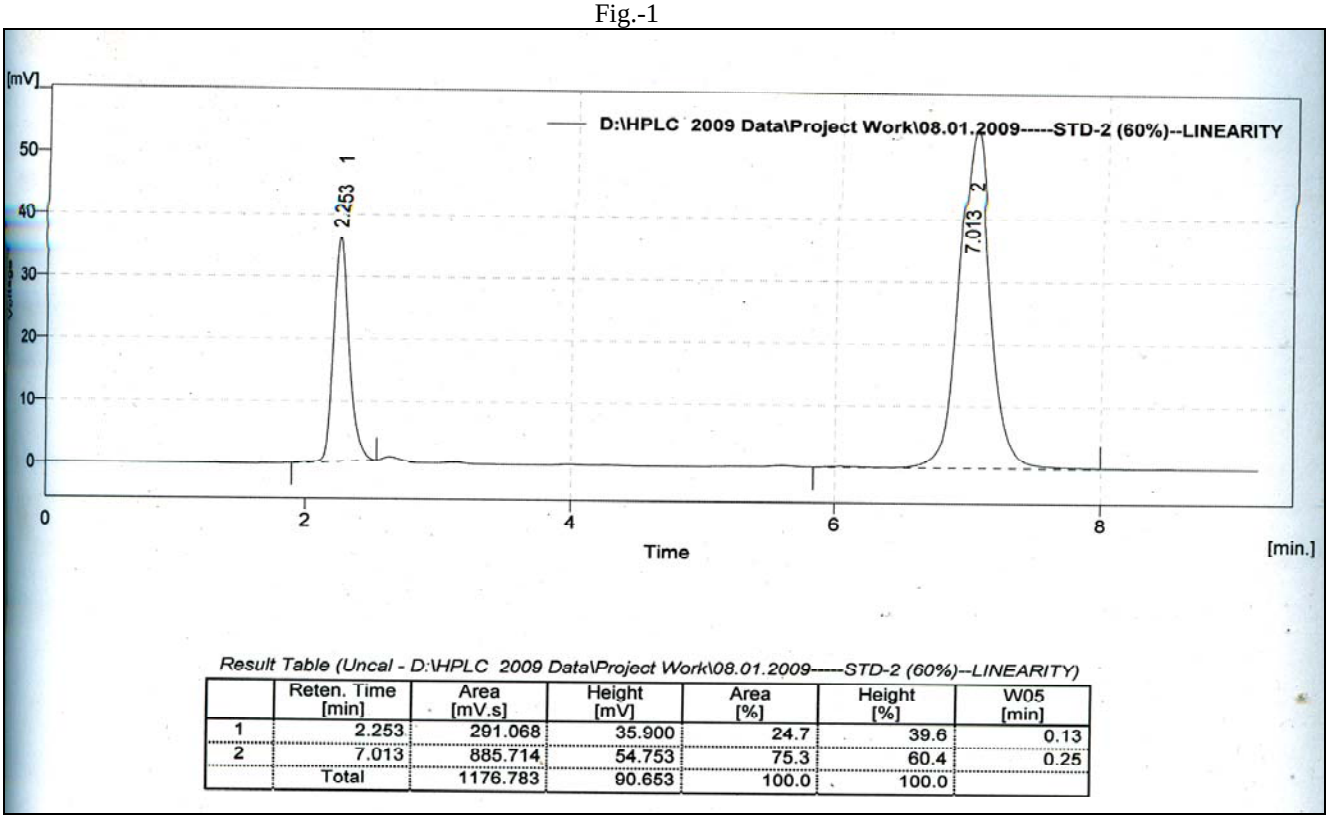


Result Table (Uncal - D:\HPLC 2009 Data\Project Work\08.01.2009-----STD-1 (SPECIFICITY))

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	2.217	505.962	62.487	25.6	40.9	0.13
2	6.777	1473.011	90.298	74.4	59.1	0.25
	Total	1978.973	152.785	100.0	100.0	

Fig.-1



Result Table (Uncal - D:\HPLC 2009 Data\Project Work\08.01.2009-----STD-2 (60%)--LINEARITY)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	2.253	291.068	35.900	24.7	39.6	0.13
2	7.013	885.714	54.753	75.3	60.4	0.25
	Total	1176.783	90.653	100.0	100.0	

Fig.-2

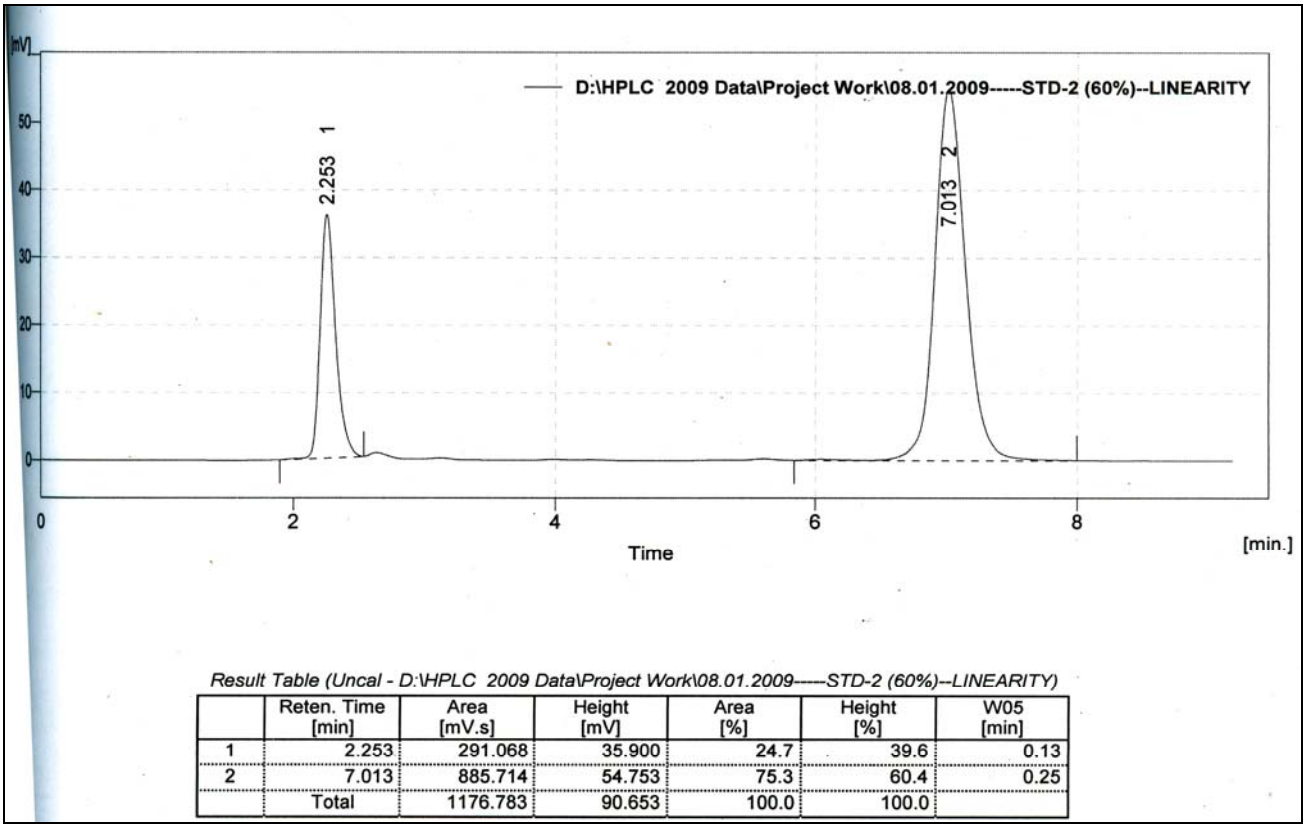


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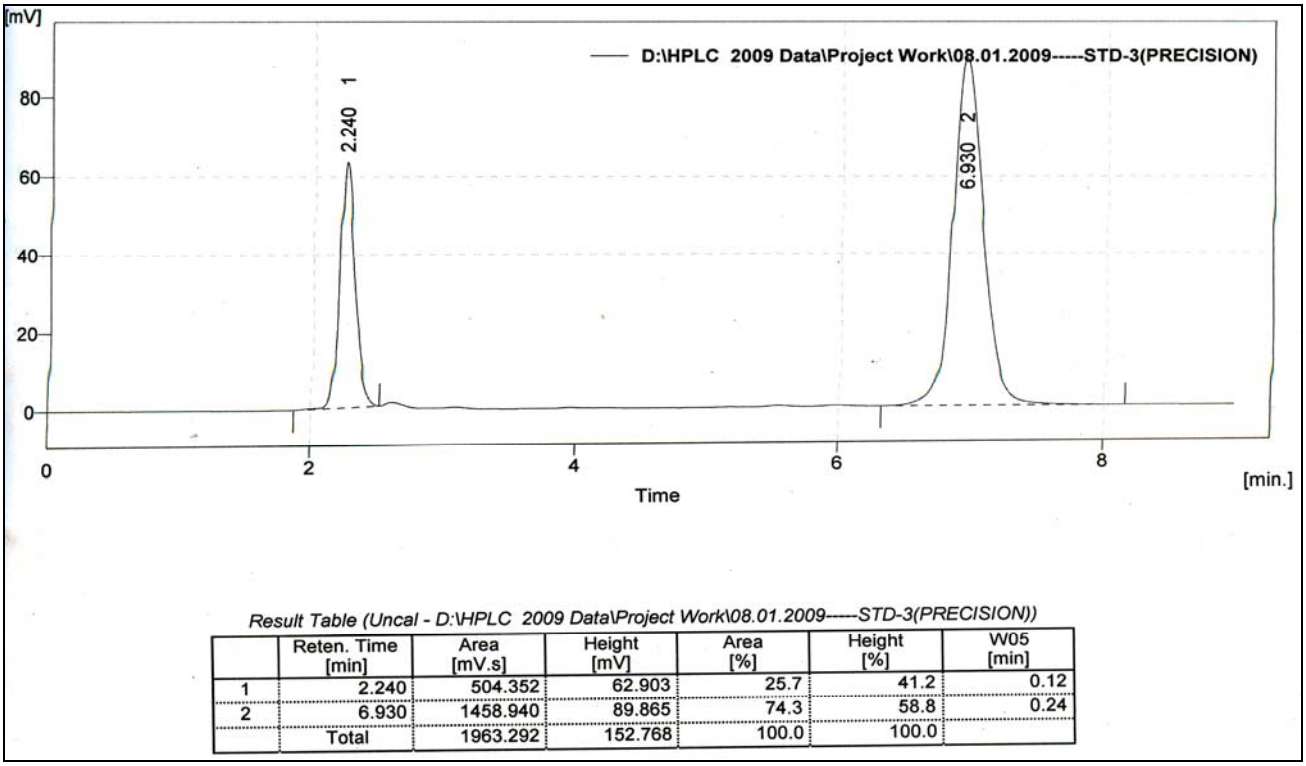
Fig.-3



Result Table (Uncal - D:\HPLC 2009 Data\Project Work\08.01.2009-----STD-3(PRECISION))

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	2.240	504.352	62.903	25.7	41.2	0.12
2	6.930	1458.940	89.865	74.3	58.8	0.24
	Total	1963.292	152.768	100.0	100.0	

Fig.-4