International Journal of Pharmacology and Clinical Research (IJPCR)

IJPCR |Vol.9 | Issue 1 | Jan - Mar -2025

www.ijpcr.com

DOI : https://doi.org/10.61096/ijpcr.v9.iss1.2025.87-96

ISSN: 2349-5448

Research

A Validation Parameters For The Simultaneous Estimation Of The Etoricoxib Tablet By Hplc Method

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Published on: 28 Feb 2025

Published by:
DrSriram Publications

Abstract

This method obeys Beers law in employed concentration range 2.4-12 µg/ml and 0.8-4 µg/ml for Pregabalin and Etoricoxib, respectively. The correlation coefficient of Etoricoxib was found to be 0.9993 and 0.9997, respectively. The retention time was 2.680 and 7.383 minutes. The percentage RSD for accuracy and precision was found to be less than 2%. The method was validated as per ICH guidelines for its selectivity, specificity, system suitability, linearity, range, precision, accuracy, LOD, LOQ, robustness, assay. The method was successfully employed for routine quality control analysis of Pregabalin and Etoricoxib in pharmaceutical formulation.

Keywords: Etoricoxib, specificity, system suitability, linearity, range, precision, accuracy, LOD, LOQ, robustness, assay

INTRODUCTION

CHROMATOGRAPHY

Generally, methods for chemical analysis are at best selective; few, if any are truly specific. Consequently the separation of the analyte from potential interferences is more often than not a vital step in analytical procedures. Amongst the most powerful techniques available to the analyst for the separation of these mixtures, a group of highly efficient methods which are collectively called as chromatography.[5, 6] It involves passing a mixture dissolved in a mobile phase through a stationary phase, which separates the analyte to be measured from other molecules in the mixture and allows it to be isolated.

Chromatography encompasses a diverse and important group of methods that permit the scientist to separate phases are chosen so that the components of the sample distribute themselves between the mobile

phase and stationary phase to varying degrees. Those phase components that are strongly retained by the stationary phase move only slowly with the flow of mobile.An active ingredient, to a number of inert materials like diluents, disintegrates, colours and Modern pharmaceutical formulations are complex mixtures containing one or more therapeutically flavours. In order to ensure quality and stability of the final product, the pharmaceutical analyst must be able to separate the mixtures into individual components prior to quantitative analysis.Chromatography may be preparative or analytical. Preparative chromatography seeks to separate the components of a mixture for further use (and is thus a form of purification). Analytical chromatography normally operates with smaller amounts of material and seeks to measure the relative proportions of analytes in a mixture. The two are not mutually exclusive.

Table 1 : Characteristics that should be considered for Different Types of Analytical Procedures (As per ICH guidelines)

S.No	Parameters	Class A	Class B		Class C	Class D
			Quantitative Tests	Limit Tests		
1.	Accuracy	-	Yes	-	Yes	Yes
2.	Precision	-	Yes	-	Yes	Yes
3.	Robustness	-	Yes	Yes	Yes	Yes
4.	Linearity and range	-	Yes	-	Yes	Yes
5.	Selectivity	Yes	Yes	Yes	Yes	Yes
6.	Limit of detection	Yes	-	Yes	-	-
7.	Limit of quantification	-	Yes	-	-	-

OBJECTIVE

Hence the present aim of my work is to develop a specific, precise, accurate, linear, simple, rapid and cost effective HPLC method for the simultaneous estimation of etoricoxib in bulk tablet dosage form.

MATERIALS AND REAGENTS

Etoricoxib working standard

Standard drug **Purity [%]**

Etoricoxib 98.71-101 % w/w

Sample used: Etoricoxib

Brand name: Arcoxia 90mg

Chemicals and solvents used

Methanol : HPLC grade

Water : HPLC grade

Acetonitrile : HPLC grade

Phosphate buffer : AR grade

Instruments used

System : HPLC Prominence / Shimadzu (Isocratic system)

Detector : UV-Visible Model SPD 20 Avp

Injector : Rheodyne

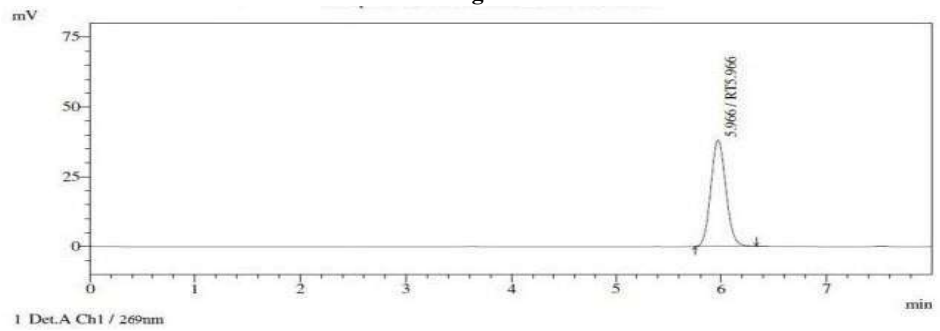
Columm : C18Phenomenex Luna

ElicoPh – Meter: LABINDIA 3000-Double beam UV / Vis spectrophotometer. Gelman Science Vacum Pump. A & D- Digital Balance.

METHOD VALIDATION

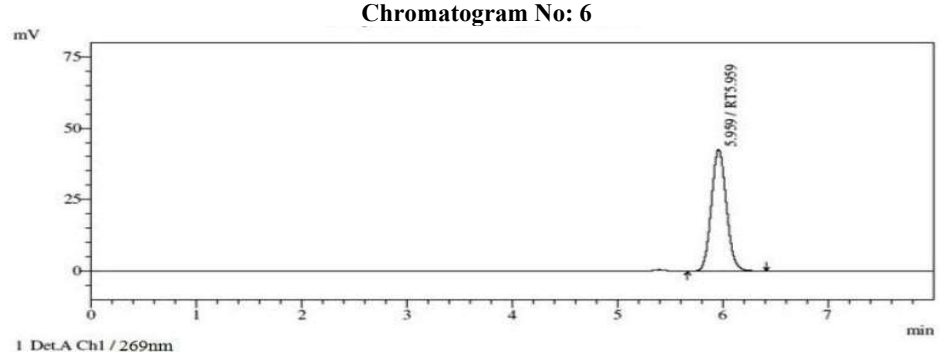
After development of HPLC method for the estimation of the multi component dosage form the validation of the method has been carried out. This section describes the procedure followed for the validation of the developed method.

Chromatogram No: 5



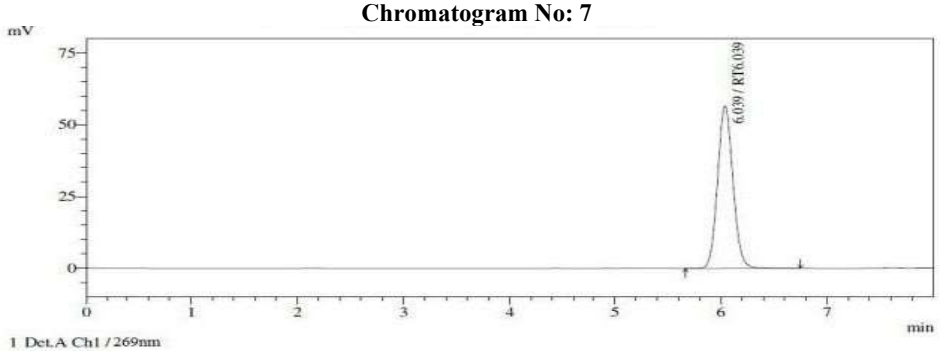
Linearity 60%

Chromatogram No: 6

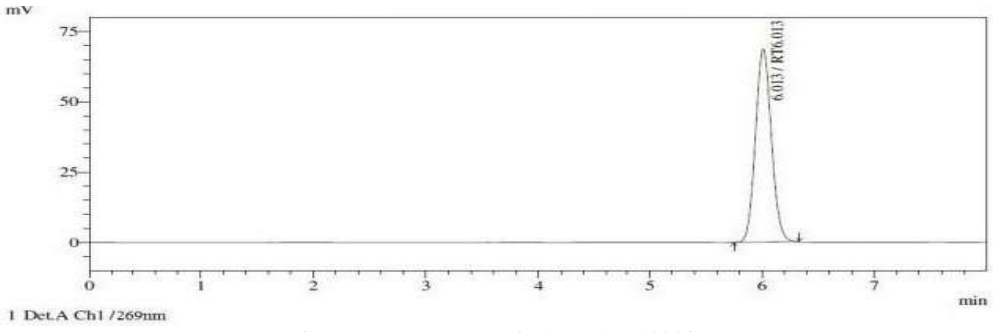


Linearity 80%

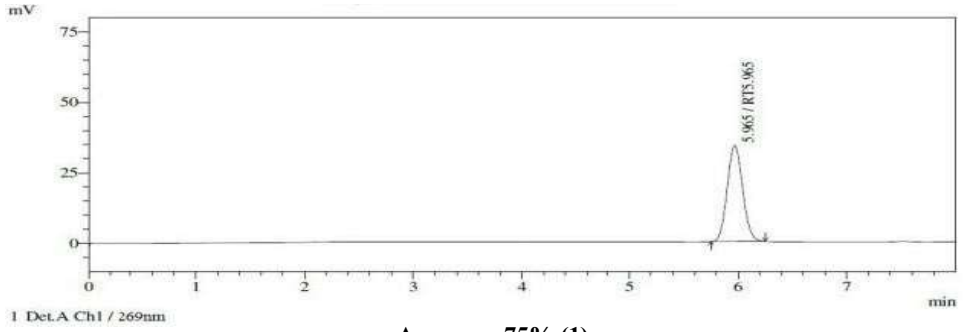
Chromatogram No: 7



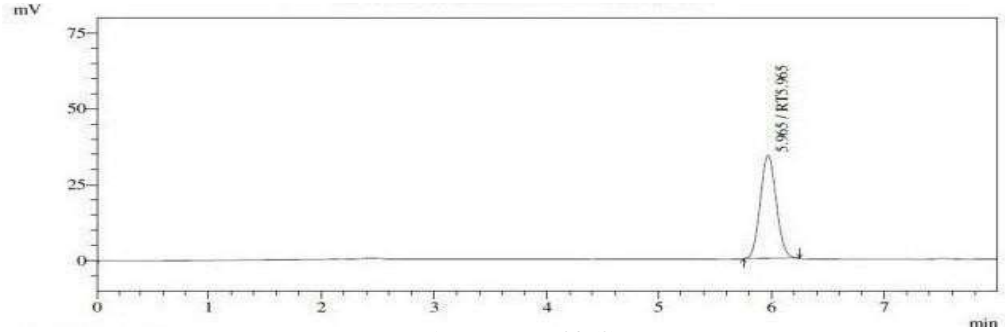
Linearity 100%



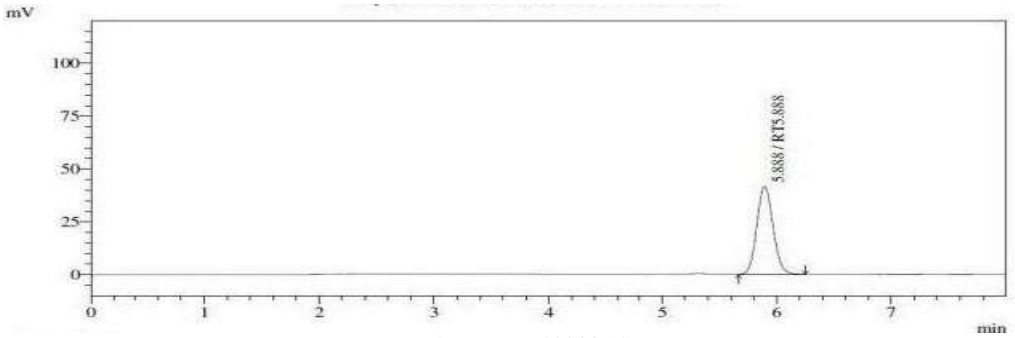
Chromatogram No: 8 Linearity 120%



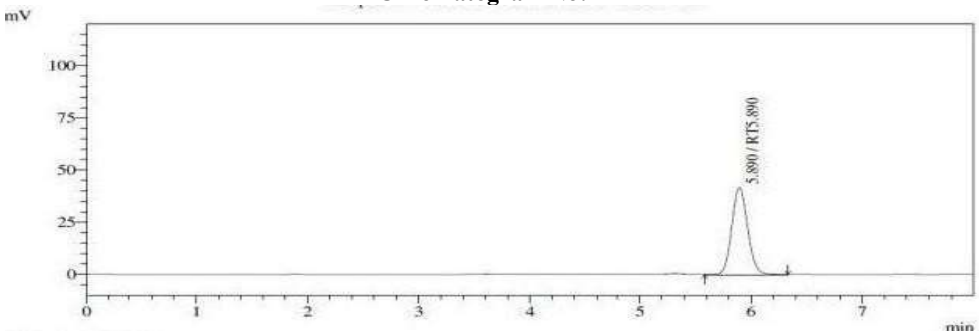
Accuracy 75% (1)
Chromatogram No: 9



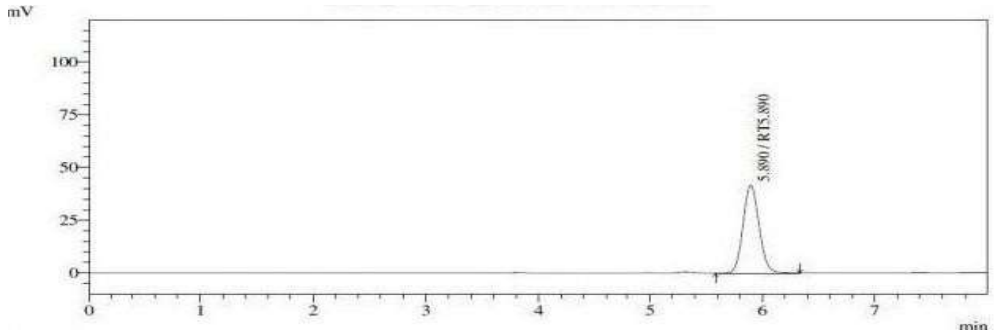
Accuracy 75% (2)
Chromatogram No: 10



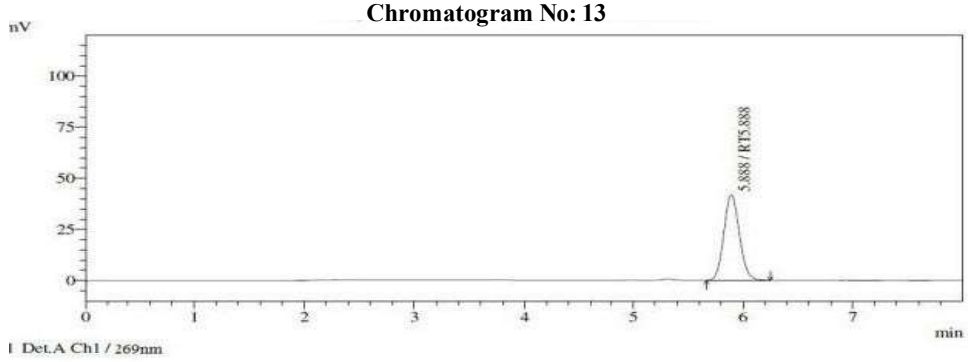
Accuracy 100% (1)
Chromatogram No: 11



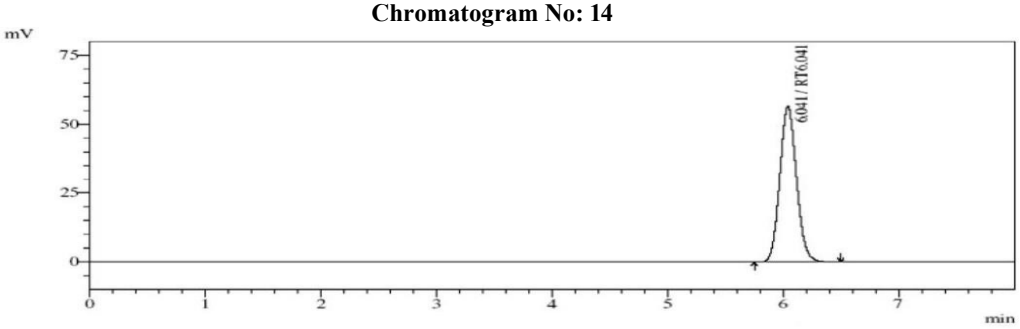
Accuracy 100% (2)
Chromatogram No: 12



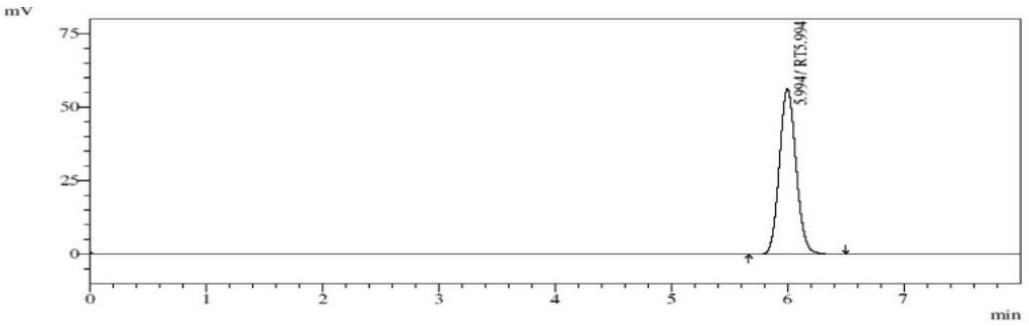
Accuracy 100% (3)
Chromatogram No: 13



Accuracy 125% (1)
Chromatogram No: 14

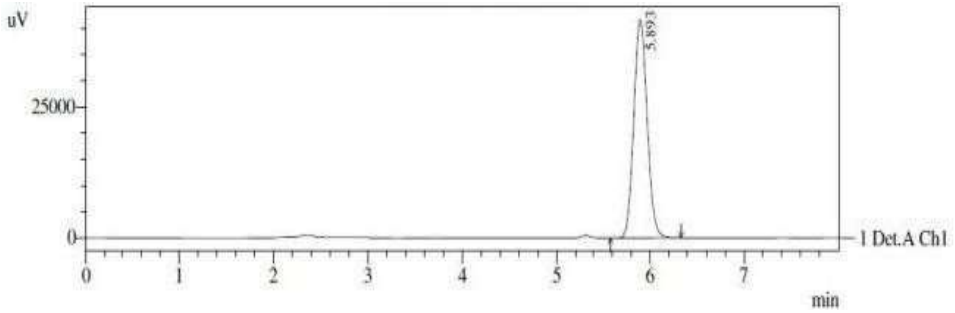


Accuracy 125% (2)
Chromatogram No: 15



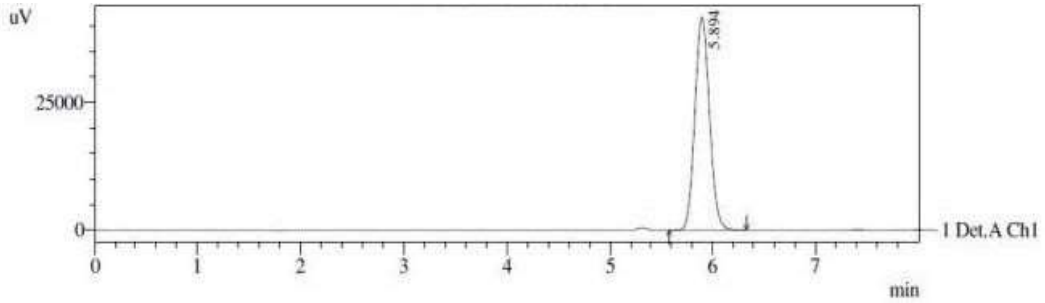
Accuracy 125% (3)

Chromatogram No: 16



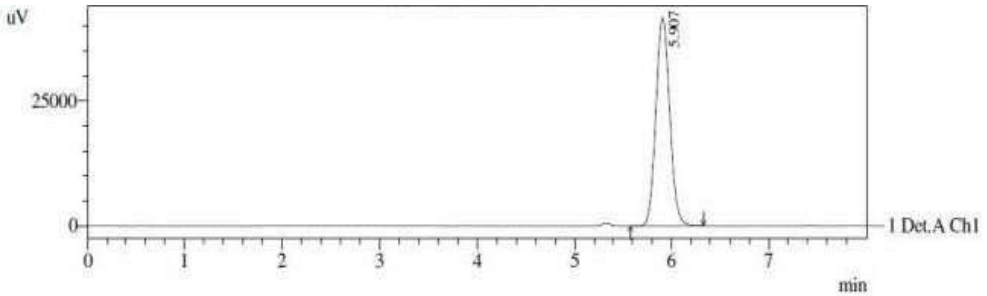
System Precission (1)

Chromatogram No: 17



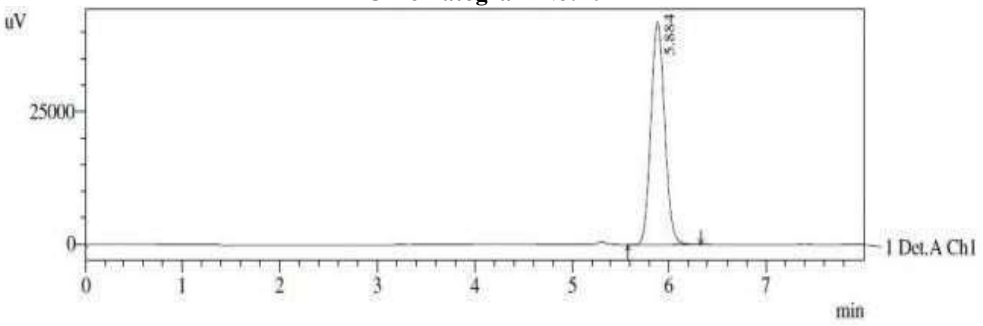
System Precission (2)

Chromatogram No: 18

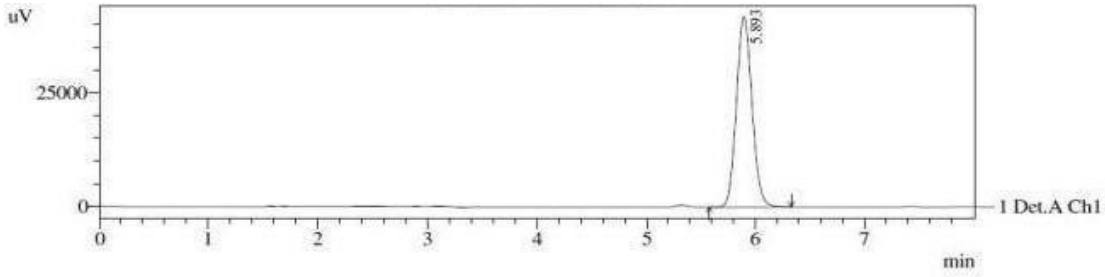


System Precission (3)

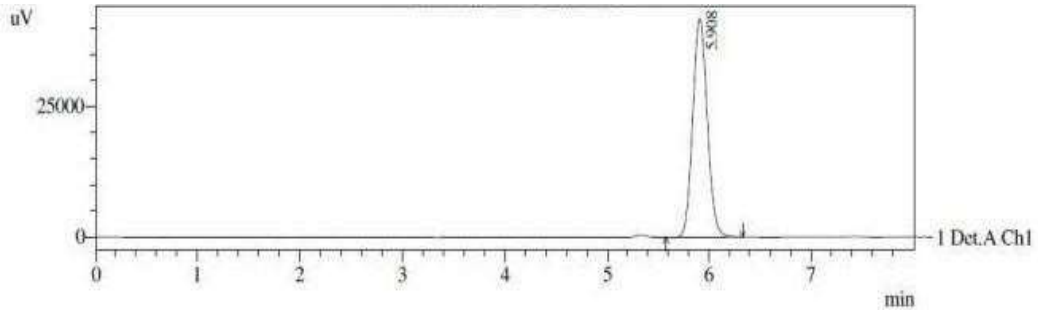
Chromatogram No: 19



System Precission (4)



Method Precission 3
Chromatogram No: 25



Method Precission 4
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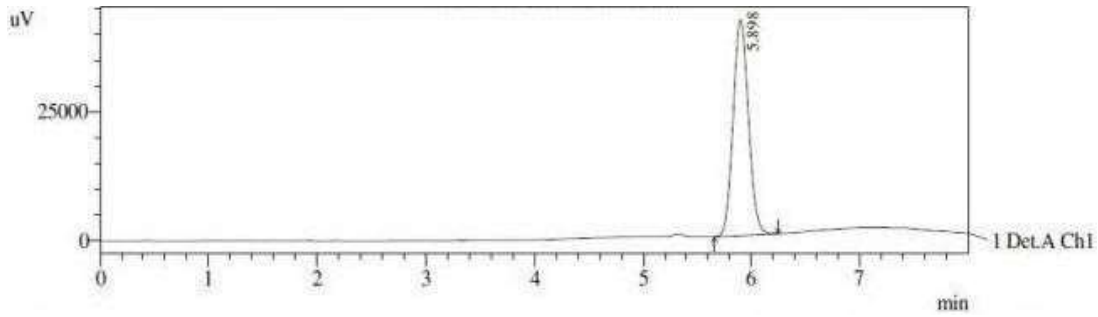
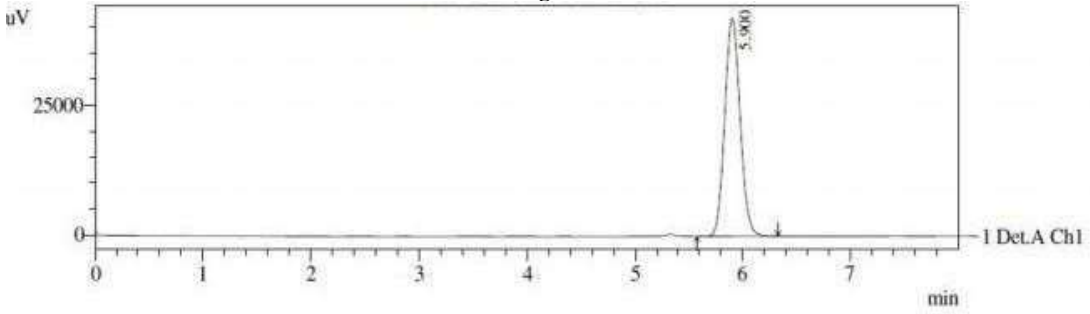
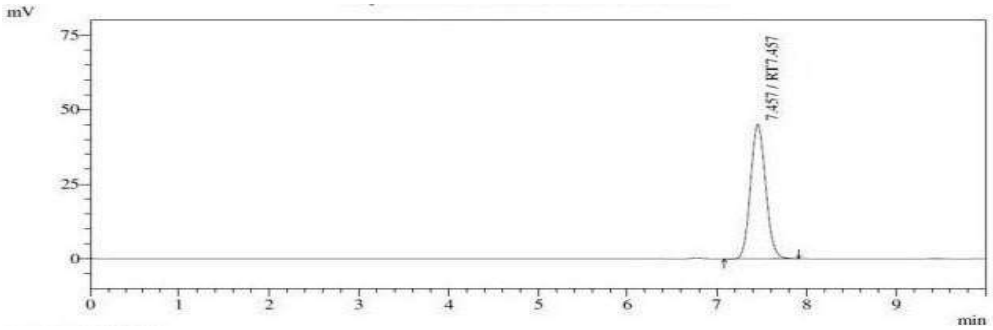


Method Precission 5
Chromatogram No: 27

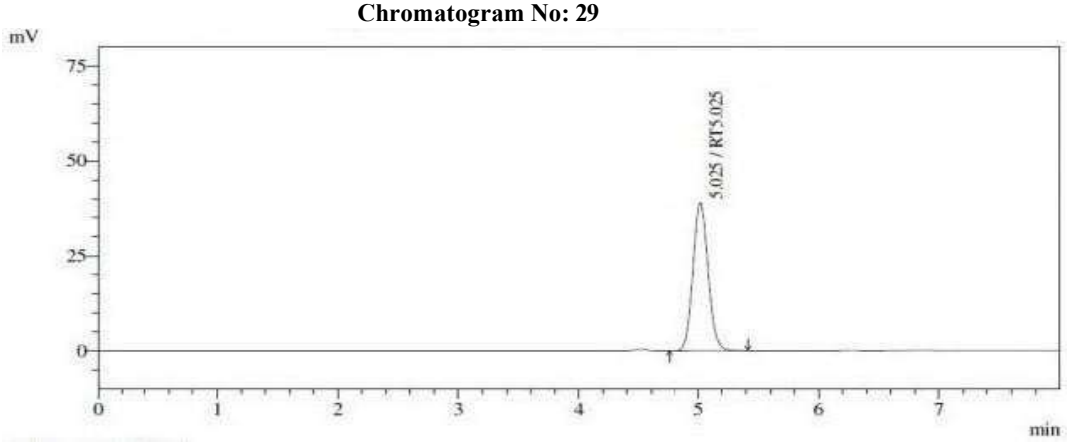


Methodprecission 6

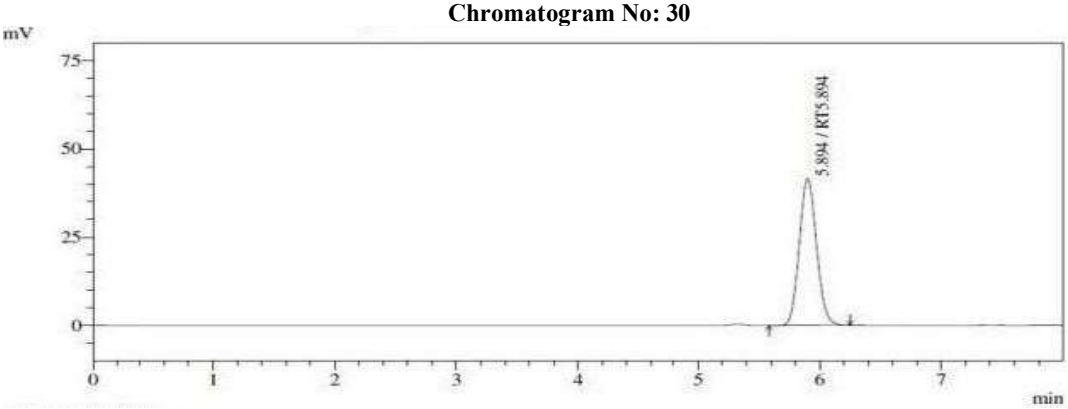
Chromatogram No: 28



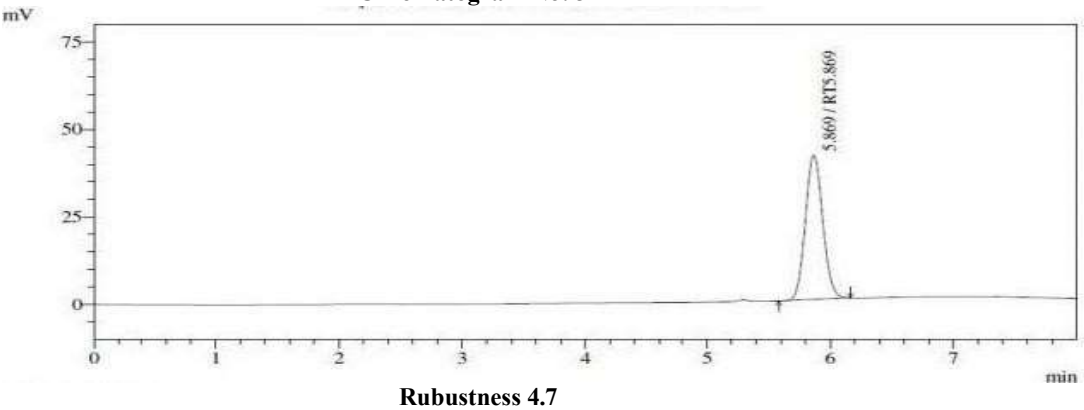
Rubustness 0.8
Chromatogram No: 29



Rubustness 1.2
Chromatogram No: 30

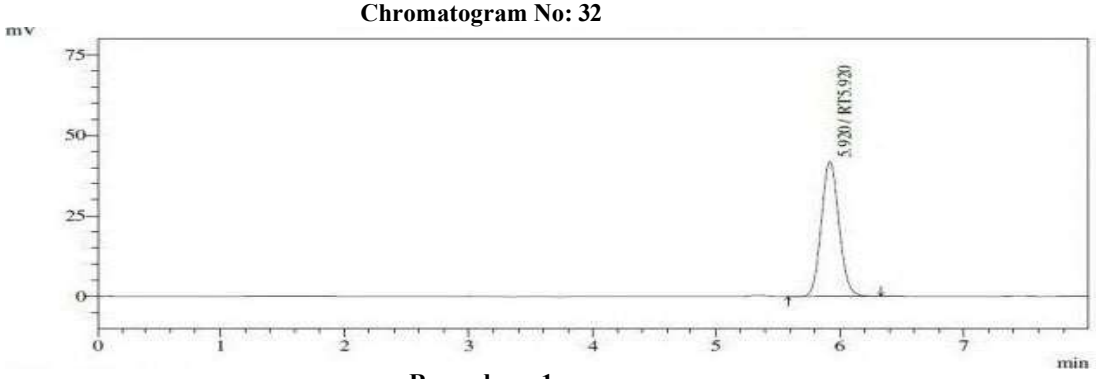


Rubustness 4.3
Chromatogram No: 31

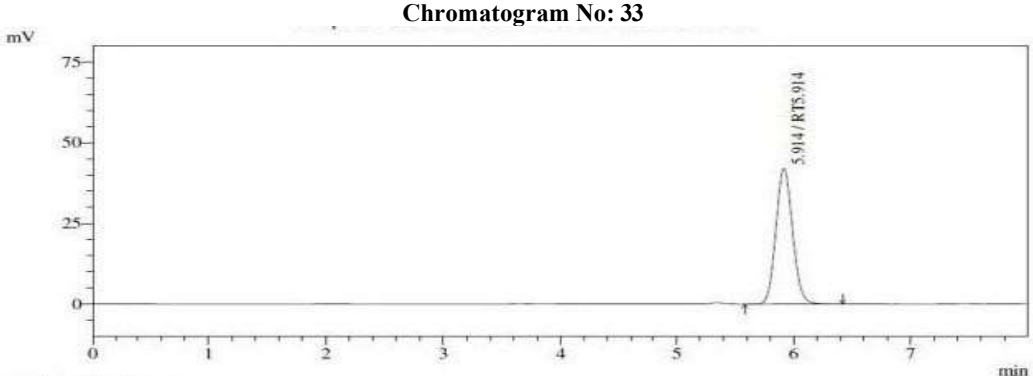


Rubustness 4.7

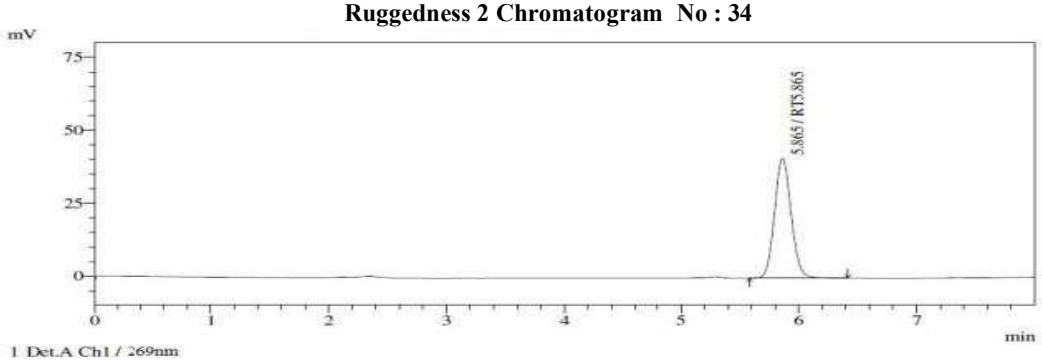
Chromatogram No: 32



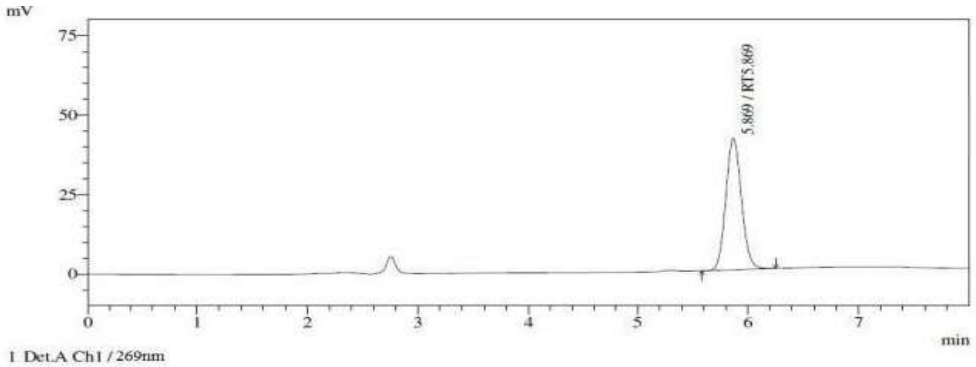
Ruggedness 1
Chromatogram No: 33



Ruggedness 2 Chromatogram No : 34



Stability Studies (0 hrs) Chromatogram No: 35



RESULTS AND DISCUSSIONS

The accuracy of the method was determined by recovery experiments. The recovery studies were carried out by preparing six individual samples with same procedure from the formulation and injecting. The percentage

recovery and percentage relative standard deviation of the percentage recovery were calculated and presented in Tables (2 and 3). From the data obtained, added recoveries of standard drugs were found to be accurate.

The precision of the method was demonstrated by system, method and repeatability of injection studies. All the solutions were injected into the chromatographic system. The peak area and percentage relative standard deviation were calculated and presented in tables (3 and 4). From the data obtained, the developed HPLC method was found to be precise.

The standard drug solutions of varying concentrations ranging from 60 % to 140 % of the targeted level of the assay concentration (i.e.) 60µg/ml to 140 µg/ml of etoricoxib were examined by the proposed method (Table 5). The response factor, slope, intercept, correlation co-efficient and Residual sum of squares values were calculated. The slope, intercept, correlation coefficient(r) were found to be 48612,

87860, 0.9991 respectively for etoricoxib. The robustness of the method was studied by carrying out experiments by changing conditions discussed earlier. The response factors for these changed chromatographic parameters were almost same as that of the fixed chromatographic parameters (table. 7 and 8) and hence developed method is said to be robust.

Conclusion: Prominence HPLC auto sampler separation module with UV detector and column used is Phenomenex – Luna C18 (250 x 4.6mm) i.d column with 5-micron particle size. Injection volume of 20□l is injected and eluted with the mobile phase of phosphate buffer: Acetonitrile: methanol (50:10:40) ratio, which is pumped at the flow rate of 1ml/min and detected by UV detector at 268.8nm.

The retention time for etoricoxib were found to be around 5.864 and 3.507 min respectively, The developed method is validated for various parameters as per ICH guidelines like accuracy, precision, linearity, specificity, system suitability, ruggedness and robustness. The results obtained are within the acceptance criteria.

The results obtained in this validation study demonstrate that the method development and validation for estimation of etoricoxib for bulk tablet dosage form by RP-HPLC, methods described is precision, specific, accurate, linear, robust and rugged with an acceptable precision. The method developed for simultaneously determination of etoricoxib for tablet dosage form were rapid, sensitive, reproducible and economical. The RP-HPLC method developed was simple and does not suffer from common excipients present in pharmaceutical preparation and highly useful in the analysis of drugs in pharmaceutical formulation.

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