

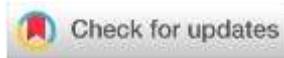


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Research Article

## Simultaneous Estimation of Pregabalin and Etoricoxib in Bulk and Pharmaceutical Dosage Form by Using RP-HPLC Method

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### Abstract

A simple, accurate, precise method was developed for the simultaneous estimation of the Pregabalin and Etoricoxib in bulk and Pharmaceutical dosage form. Chromatogram was run through Ascentis (C18 150 x 4.8 mm, 2.8m). Mobile phase containing Buffer 0.01N Methanol: Na2HPO4 (0.1%OPA is added to adjust pH 5.4) taken in the ratio 70:30 was pumped through column at a flow rate of 1ml/min. Temperature was maintained at 30°C. Optimized wavelength selected was 235nm. Retention time of Pregabalin and Etoricoxib were found to be 2.396 minutes and 2.968 minutes. %RSD of the Pregabalin and Etoricoxib were found to be 1.0 and 1.1 respectively. %Recovery was obtained as 99.82% and 99.99% for Pregabalin and Etoricoxib respectively. LOD, LOQ values obtained from regression equations of Pregabalin and Etoricoxib were 0.27, 0.81 and 0.18, 0.55 respectively. Regression equation of Pregabalin is  $y=3501.2x+839.82$ , and  $y=3696.3x+1383.5$  for Etoricoxib. Retention time and run time was decreased, so the method was developed simple and economical that can be adopted in regular Quality control test.

**Keywords:** Pregabalin, Etoricoxib, RP-HPLC, Method validation

## INTRODUCTION

The quality of a drug plays an important role in ensuring the safety and efficacy of the drugs. Quality assurance and control of pharmaceutical and chemical formulations is essential for ensuring the availability of safe and effective drug formulations to consumers. Hence analysis of pure drug substances and their pharmaceutical dosage forms occupies a pivotal role in assessing the suitability to use in patients. The quality of the analytical data depends on the quality of the methods employed in generation of the data<sup>1</sup>. Hence, development of rugged and robust analytical methods is very important for statutory certification of drugs and their formulations with the regulatory authorities.

The quality and safety of a drug is generally assured by monitoring and controlling the assay and impurities effectively. While assay determines the potency of the drug and impurities will determine the safety aspect of the drug. Assay of pharmaceutical products plays an important role in efficacy of the drug in patients.

The wide variety of challenges is encountered while developing the methods for different drugs depending on its nature and properties. Along with this importance of achieving the selectivity, speed, cost, simplicity, sensitivity, reproducibility and accuracy of results gives an opportunity

for researchers to come out with solution to address the challenges in getting the new methods of analysis to be adopted by the pharmaceutical industry and chemical laboratories. Different physico-chemical methods<sup>1</sup> are used to study the physical phenomenon that occurs as a result of chemical reactions. Among the physico-chemical methods, the most important are optical (refractometry, polarimetry, emission and fluorescence methods of analysis), photometry (photocolorimetry and spectrophotometry covering UV-Visible, IR spectroscopy and nephelo turbidometry) and chromatographic (column, paper, thin layer, gas liquid and high performance liquid chromatography) methods. Methods such as nuclear magnetic resonance (NMR) and para magnetic resonance (PMR) are becoming more and more popular. The combination of mass spectroscopy (MS) with gas chromatography is one of the most powerful tools available. The number of new drugs is constantly growing. This requires new methods for controlling their quality. Modern pharmaceutical analysis must need the following requirements.

1. The analysis should be took a minimal time.
2. The accuracy of the analysis should be meet the demands of Pharmacopoeia.
3. The analysis should be economical.
4. The selected method should be precise and selective.

## DRUG PROFILE

### Pregabalin

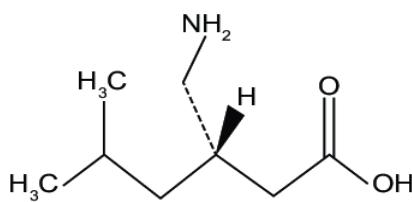


Fig1: Structure of Pregabalin

### Physicochemical properties

**Chemical Formula:** C8H17NO2

**IUPAC Name:** (3S)-3-(amino methyl)-5-methylhexanoic acid

**Molar Mass:** 159.23 g/mol

**Melting Point:** 176-178° C

**Boiling Point:** 144-147° C

**Solubility:** Freely soluble, Water solubility-11.3 mg/mL

Pregabalin is an anticonvulsant medication used in combination with other anticonvulsant medications to treat partial onset seizures, fibromyalgia, and neuropathic pain problems. It exhibits structural similarities with the inhibitory neurotransmitter gamma amino butyric acid (GABA).

### Etoricoxib

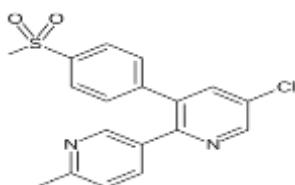


Fig2: Structure of Etoricoxib

### Physicochemical properties

**Chemical Formula:** C18H15ClN2O2S

**IUPAC Name:** 5-chloro-3-(4-methanesulfonylphenyl)-6'-methyl-2,3'-bipyridine

**Molar Mass:** 358.842 g/mol

**Boiling Point:** 510° C

**Solubility:** Water solubility-0.00328 mg/mL

Etoricoxib is selective COX-2 inhibitor used to treat various types of inflammatory and painful arthritic symptoms as well as moderate post-surgical dental pain. Treatment of rheumatoid arthritis, osteoarthritis, ankylosing spondylitis, chronic low back pain, acute pain, and gout are among the current therapeutic indications.

## MATERIALS AND METHODS

### Materials

Pregabalin and Etoricoxib pure drugs (API), Combination Pregabalin and Etoricoxib tablets (EMAXGALIN), Distilled water, Acetonitrile, Phosphate buffer, Methanol, Potassium dehydrogenate ortho phosphate buffer, Ortho-phosphoric acid.

### Methods

**Diluent:** Based up on the solubility of the drugs, diluent was selected, Acetonitrile and Water taken in the ratio of 50:50

**Preparation of Standard stock solutions:** 30 mg of Etoricoxib and 37.5 mg of Pregabalin was separately weighed

and transferred into 50 ml volumetric flasks. Both of these flasks received 3/4 th of diluents, which were then sonicated for 10 minutes which gives 750 $\mu$ g/ml of Pregabalin and 600 $\mu$ g/ml of Etoricoxib solution.

**Preparation of Sample stock solutions:** The combination powder sample was accurately weighed and transferred into a 100 ml volumetric flask. 50 ml of diluents were added, and the mixture was sonicated for 25 minutes. Further, the volume was made up with diluent and filtered through milli-Q filters which gives 750 $\mu$ g/ml of Pregabalin and 600 $\mu$ g/ml of Etoricoxib solution. From that working standard 75 and 60 $\mu$ g/ml was prepared.

### Preparation of buffer:

**0.1% OPA Buffer:** 1 ml of concentrated Ortho Phosphoric acid was diluted to 1000 ml with water.

**0.01N NaHPO4 Buffer:** Accurately weighed 1.42 gm of Potassium dihydrogen Ortho phosphate in a 1000 ml of Volumetric flask add about 900 ml of milli-Q water added and sonicate and finally make up the volume with water then pH adjusted to 4.0 with dil. Ortho phosphoric acid solution.

### Validation

#### System suitability parameters:

The standard solutions of Pregabalin (75 ppm) and Etoricoxib (60 ppm) were prepared, and the solutions were injected six times to determine parameters like peak tailing, resolution, and USP plate count.

The % RSD for the area of six standard injections results should not be more than 2%.

**Specificity:** Checking for interference in a method that has been optimized. At the retention times of these drugs in this approach, we should not detect interference peaks in blank and placebo samples. So, it was stated that this approach was specific.

**Precision:** Precision of the data was reported in terms of Repeatability, Intra-day precision, and Inter-day precision. The %RSD values were calculated. The results indicate that the method is precise.

**Linearity:** The linearity of the method was estimated by preparing calibration samples which were prepared by spiking appropriate amount of Pregabalin and Etoricoxib in water and acetonitrile to give 0-112.5 $\mu$ g/mL and 0-90  $\mu$ g/mL.

**Limit of detection (LOD) and Limit of quantification (LOQ):** The sensitivity of the proposed method for measurement of pregabalin and etoricoxib was estimated in terms of LOD & LOQ. The Limit of detection (LOD) and limit of quantification (LOQ) was determined according to the ICH guidelines for the validation of analytical procedure. The formula used was:

$$LOD = 3.3\sigma/S$$

$$LOQ = 10\sigma/S$$

Where,  $\sigma$  = standard deviation of the response (intercept)

$S$  = slope of the calibration curve

**Accuracy:** Accuracy of the method was determined at three different levels (50%, 100%, and 150%) mean and %RSD values were calculated. The results indicate that the method was accurate.

**Robustness:** Small deliberate adjustments were made to the procedure, such as flow rate, mobile phase ratio, and temperature, but there was no noticeable variance in the outcome and it remained within the ICH guideline range.

Flow minus (0.9 ml/min), Flow plus (1.1 ml/min), mobile phase minus (25°C), mobile phase plus (35°C), and temperature minus (25°C) and temperature plus (35°C) robustness conditions were maintained, and samples were injected in duplicate. The parameters for system suitability were not significantly impacted, and all of the parameters were met. The % RSD for the area should not be more than 2%.

#### Degradation studies

**Oxidation:** 1 ml of 20% hydrogen peroxide was added separately to 1 ml of the stock solution of Pregabalin and Etoricoxib. The solutions were maintained at 60° C for 30 minutes. In order to determine the sample's stability for the HPLC study, the resulting solution was diluted to obtain 75 µg/ml and 60 µg/ml solutions. 10 µl were then injected into the system and the chromatograms were recorded.

**Acid Degradation Studies:** 1ml of the stock solution of pregabalin and etoricoxib was combined with 1ml of 2N hydrochloric acid, which was then refluxed for 30 minutes at 60° C. The resulting solution was diluted to obtain 75µg/ml and 60µg/ml solutions, and 10 µl solutions were injected into the system. Chromatograms were recorded to determine the sample's stability.

**Alkali Degradation Studies:** 1ml of 2N sodium hydroxide was added to 1 ml of stock solution of pregabalin and etoricoxib, which was refluxed for 30 minutes at 60°C. The resulting solution was diluted to obtain 75 µg/ml and 60 µg/ml solutions, and 10 µl were injected into the system. The chromatograms were recorded to determine the sample's stability.

**Dry Heat Degradation Studies:** To evaluate dry heat degradation, the standard drug solution was heated to 105°C for one hour. The final solution was diluted to a concentration

of 75µg/ml and 60µg/ml and 10µl solutions were injected into the apparatus for HPLC study. Chromatograms were recorded to determine the sample's stability.

**Photo Stability studies:** By keeping the beaker in a UV chamber for 1 day or 200 Watt hours/m<sup>2</sup> in a photochemical stability chamber, the 75µg/ml and 60µg/ml solutions were exposed to UV light in order to study the drug's photochemical stability. The final solution was diluted to obtain 75 µg/ml and 60 µg/ml solutions for the HPLC analysis. 10 µl were injected into the system, and the chromatograms were recorded to determine the sample's stability.

**Neutral Degradation Studies:** The drug was refluxed in water for one hour at a temperature of 60° C to study stress testing in neutral conditions. To determine the stability of the sample, the final solution was diluted to 75µg/ml and 60µg/ml solution, 10 µl were injected into the system, and the chromatograms were recorded.

## RESULTS AND DISCUSSION

**Method development:** Method development was done by changing various mobile phase ratios, buffers etc.

**Optimized Chromatogram:** The chromatography elution was carried out in the isocratic mode where the mobile phase was selected as methanol and phosphate buffer (pH 4.0 adjusted with dil. Ortho phosphoric acid) in ratio (80:20). The flow rate of the mobile phase was selected as 1 ml/min with a run time of 7 minutes at ambient temperature. The eluent was passed through Ascentis C18 column (4.8 x 150mm, 2.8µm) and monitoring by using uv detector at 235nm.

**System suitability:** All the system suitability parameters were within the range and satisfactory as per ICH guidelines

**Table 1: System suitability parameters for Pregabalin and Etoricoxib**

Pregabalin			Etoricoxib			
RT (min)	USP Plate Count	Tailing	RT (min)	USP Plate Count	Tailing	Resolution
2.364	8673	1.2	2.92	11282	1.18	5.1
2.365	8794	1.2	2.921	10709	1.19	5.1
2.365	8671	1.19	2.923	10534	1.21	5.2
2.365	8999	1.2	2.923	10502	1.23	5.2
2.366	9010	1.2	2.923	10531	1.23	5.2
2.366	8980	1.21	2.931	10788	1.17	5.3

**Specificity:** Pregabalin and Etoricoxib had retention times of 2.396 minutes and 2.968 minutes, respectively. In the blank and placebo, we did not detect any interfering peaks at the

respective drug retention times using this method. So, it was claimed that this method was specific.

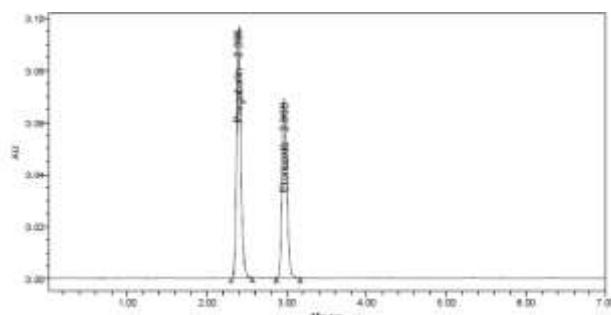


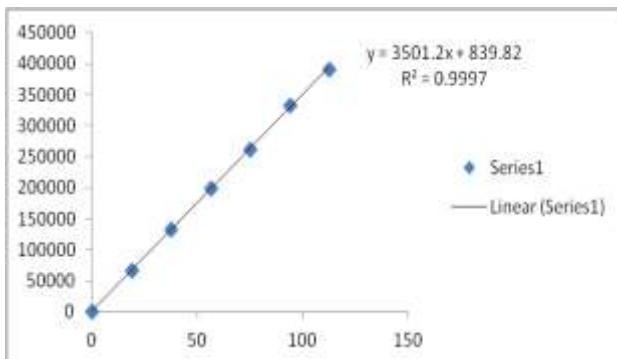
Figure 3: Typical chromatogram

**Linearity:** Six linear concentrations of Pregabalin (18.75-112.5 $\mu$ g/ml) and Etoricoxib (15-60 $\mu$ g/ml) were injected in a duplicate manner. Average areas were mentioned above and

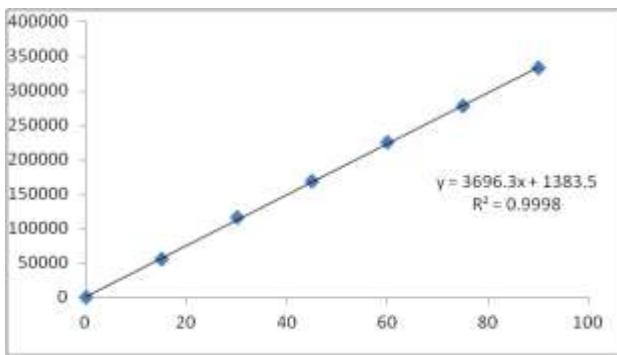
linearity equations obtained for Pregabalin was  $y=3501.2x+839.82$  and of Etoricoxib was  $y=3696.3x+1383.5$ . Correlation coefficient obtained was 0.999 for the two drugs.

**Table 2: Linearity table for Pregabalin and Etoricoxib**

Pregabalin		Etoricoxib	
Concentration ( $\mu$ g/mL)	Peak area	Concentration ( $\mu$ g/mL)	Peak area
0	0	0	0
18.75	66199	15	55508
37.5	132356	30	115433
56.25	198694	45	167643
75	262549	60	224949
93.75	333695	75	277492
112.5	390980	90	332981



**Figure 4: Calibration curve of Pregabalin**



**Figure 5: Calibration curve of Etoricoxib**

#### Sensitivity:

**Table 3: Sensitivity table of Pregabalin and Etoricoxib**

Molecule	LOD	LOQ
Pregabalin	0.27	0.81
Etoricoxib	0.18	0.53

#### Precision:

**Repeatability:** From a single volumetric flask of working standard solution, six injections were given and the obtained areas were mentioned above. Average area, standard deviation and % RSD were calculated for two drugs. % RSD obtained as 0.5% and 0.7% respectively for Pregabalin and Etoricoxib. As the limit of precision was less than "2" the Method Precision was passed in this method.

**Table 4: Repeatability table of Pregabalin and Etoricoxib**

S. No.	Area of Pregabalin	Area of Etoricoxib
1.	265870	221269
2.	264123	225313
3.	266059	223326
4.	264270	225080
5.	263660	224531
6.	262497	225142
<b>Mean</b>	<b>264413</b>	<b>224110</b>
<b>S.D</b>	<b>1354.6</b>	<b>1569.5</b>
<b>%RSD</b>	<b>0.5</b>	<b>0.7</b>

**Intermediate precision (Day-Day Precision):** Six working sample solutions of the same concentrations were prepared after performing several samplings from a sample stock solution. Each injection from a working sample solution was administered the next day after the sample preparation, and the results were listed in the table 5. For two drugs, average area, standard deviation, and percent RSD were calculated; the results were 0.9% and 0.9% for Pregabalin and Etoricoxib, respectively. The system precision was passed using this method since the precision limit was less than "2".

**Table 5: Intermediate precision (Day-Day Precision) table of Pregabalin and Etoricoxib**

S. No.	Area of Pregabalin	Area of Etoricoxib
1.	265880	224898
2.	262392	228139
3.	262782	226724
4.	268539	226943
5.	263538	222110
6.	264427	226659
<b>Mean</b>	<b>264593</b>	<b>225912</b>
<b>S.D</b>	<b>2301.8</b>	<b>2131.8</b>
<b>%RSD</b>	<b>0.9</b>	<b>0.9</b>

**Accuracy:** Three levels of Accuracy samples were prepared by standard addition method. Triplicate injections were given for each level of accuracy and mean %Recovery was obtained as

99.82% and 99.99% for Pregabalin and Etoricoxib respectively.

**Table 6: Accuracy table of Pregabalin**

% Level	Amount Spiked (µg/mL)	Amount Recovered (µg/mL)	% Recovery	Mean % Recovery
50%	37.5	37.47	99.93	99.82%
	37.5	37.68	100.49	
	37.5	37.60	100.27	
100%	75	75.38	100.51	99.82%
	75	73.39	97.85	
	75	73.84	98.45	
150%	112.5	113.46	100.85	99.82%
	112.5	113.14	100.57	
	112.5	111.81	99.41	

**Table 7: Accuracy table of Etoricoxib**

% Level	Amount Spiked (µg/mL)	Amount Recovered (µg/mL)	% Recovery	Mean % Recovery
50%	30	29.85	99.51	99.99%
	30	30.20	100.68	
	30	30.02	100.07	
100%	60	60.34	100.57	99.99%
	60	59.44	99.07	
	60	60.10	100.16	
150%	90	90.33	100.36	99.99%
	90	90.12	100.13	
	90	89.40	99.33	

**Robustness:** Conditions for robustness including Flow minus (0.9ml/min), Flow plus (1.1ml/min), mobile phase minus (65B:35A), mobile phase plus (75B:25A), temperature minus (27°C) and temperature plus(33°C) was maintained and

samples were injected in duplicate. The parameters for system suitability were not significantly affected, and all the parameters were met. %RSD was within the permitted range.

**Table 8: Robustness data for Pregabalin and Etoricoxib.**

S. No.	Condition	%RSD of Pregabalin	%RSD of Etoricoxib
1.	Flow rate (-) 0.9 ml/min	0.7	0.8
2.	Flow rate (+) 1.1 ml/min	0.8	1.2
3.	Mobile phase (-) 65B:35A	1.7	1.3
4.	Mobile phase (+) 75B:25A	0.8	0.5
5.	Temperature (-) 27° C	0.9	1.4
6.	Temperature (+) 33° C	0.6	0.4

**Assay:** (EMAXGALIN), bearing the label claims Pregabalin 75mg, Etoricoxib 60mg. Assay was performed with the above

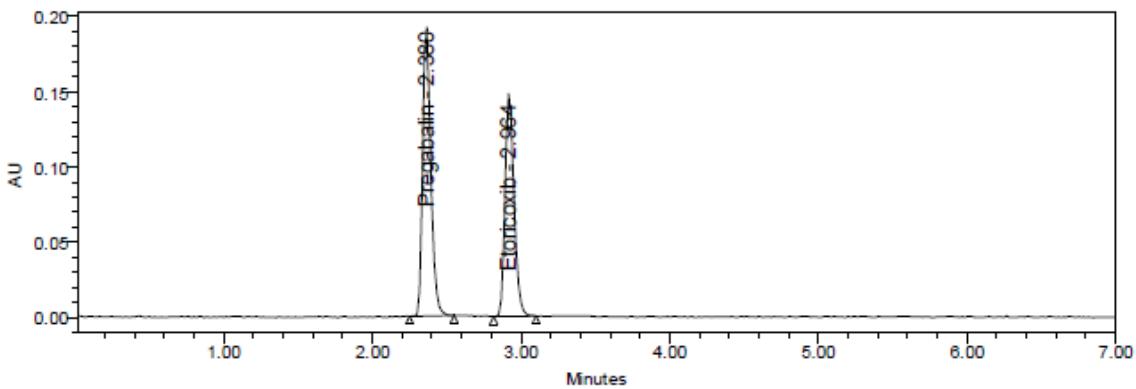
formulation. Average % Assay for Pregabalin and Etoricoxib obtained was 99.84% and 100.45% respectively.

**Table 9: Assay Data of Pregabalin**

S.No	Standard Area	Sample area	% Assay
1	265649	265870	100.39
2	264101	264123	99.73
3	266741	266059	100.46
4	265026	264270	99.79
5	266578	263660	99.56
6	259331	262497	99.12
<b>Avg</b>	264571	264413	99.84
<b>S.D</b>	2749.0	1354.6	0.51
<b>%RSD</b>	1.0	0.5	0.5

**Table 10: Assay Data of Etoricoxib**

S.No	Standard Area	Sample area	% Assay
1	221405	221269	99.18
2	224581	225313	100.99
3	225574	223326	100.10
4	224936	225080	100.89
5	221448	224531	100.64
6	219325	225142	100.91
<b>Avg</b>	222878	224110	100.45
<b>S.D</b>	2499.7	1569.5	0.7035
<b>%RSD</b>	1.1	0.7	0.7



**Figure 6: Chromatogram of working sample solution**

#### Degradation data:

**Table 11: Degradation data for Pregabalin and Etoricoxib**

Type of degradation	Pregabalin			Etoricoxib		
	Area	% Recovered	% Degraded	Area	% Recovered	% Degraded
Acid	255128	96.33	3.67	218214	97.81	2.19
Base	257404	97.19	2.81	217305	97.40	2.60
Peroxide	247644	93.51	6.49	210198	94.22	5.78
Thermal	259515	97.99	2.01	220523	98.84	1.16
UV	260057	98.20	1.80	221737	99.39	0.61
Water	262763	99.22	0.78	221633	99.34	0.66

**SUMMARY**

Parameters	Pregabalin	Etoricoxib	Limit
<b>Linearity Range (µg/ml)</b>	18.75-112.5µg/ml	15- 90µg/ml	R< 1
<b>Regression coefficient</b>	0.999	0.999	
<b>Slope(m)</b>	3501.2	3696.3	
<b>Intercept(c)</b>	839.82	1383.5	
<b>Regression equation (Y=mx+c)</b>	y = 3501.2x + 839.82	y = 3696.3x + 1383.5	
<b>Assay (% mean assay)</b>	100.42%	100.45 %	90-110%
<b>Specificity</b>	Specific	Specific	No interference of any peak
<b>System precision %RSD</b>	1.0	1.1	NMT 2.0%
<b>Method precision %RSD</b>	0.5	0.7	NMT 2.0%
<b>Accuracy %recovery</b>	99.82%	99.99%	98-102%
<b>LOD</b>	0.27	0.18	NMT 3
<b>LOQ</b>	0.81	0.55	NMT 10
<b>Robustness</b>	<b>FM</b>	0.7	0.8
	<b>FP</b>	0.8	1.2
	<b>MM</b>	1.7	1.3
	<b>MP</b>	0.8	0.5
	<b>TM</b>	0.9	1.4
	<b>TP</b>	0.6	0.4

**CONCLUSION**

HPLC method was developed and validated as per ICH guidelines. The proposed method for the assay of Pregabalin and Etoricoxib tablets or capsules is very simple and rapid. It should be emphasized that it is isocratic and the ease of mobile phase facilitated better elution of the drugs. The method was validated for specificity, linearity, precision, accuracy and robustness. The present developed can be effectively used for the routine analysis of Pregabalin and Etoricoxib in pharmaceutical formulations.

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**CONFLICTS OF INTEREST**

There are no conflicts of interest.

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