

# Validated Stability Indicating FT-IR Spectroscopic Method For Simultaneous Quantitative Estimation Of Aceclofenac And Pregabalin In Combined Tablet Dosage Form

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## Abstract

The current study focused on FT-IR spectroscopic method development and validation for simultaneous estimation of aceclofenac (ACF) and pregabalin (PGL) in combined tablet dosage form. Aceclofenac and pregabalin are used in combination for the relief of pain and inflammation in rheumatoid arthritis, osteoarthritis, and ankylosing spondylitis and adjunctive treatment of partial seizure, epilepsy, fibromyalgia, neuropathic pain drug. Literature survey revealed that no method is reported on FTIR method for the simultaneous estimation of the aceclofenac & pregabalin in bulk and combined pharmaceutical dosage form. The method is validated with respect to linearity, precision, accuracy, robustness, ruggedness and system suitability parameters as per ICH guidelines. The developed method was applied to forced degradation studies successfully to verify the utility of the established procedure.

**Keywords:** Aceclofenac, Pregabalin, Forced degradation studies, FT-IR, Validation and ICH.

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## 1. Introduction

Fourier transform Infrared spectroscopy (FTIR) spectroscopy is one of the most common spectroscopic techniques used by organic and inorganic chemists. Simply, it is the absorption

measurement of different IR frequencies by a sample positioned in the path of an IR beam. The main goal of IR spectroscopic analysis is to determine the chemical functional groups in the sample. Fourier transform infrared spectroscopy is simple, rapid, solvent free, economic and eco-friendly technique which plays a major role for the rapid determination of various components present in the sample as well as complex matrices. In pharmaceutical chemistry FTIR is widely used for the identification of compounds, impurities and determination of functional groups for qualitative analysis of pharmaceuticals and drug in liquid or solid form. Initially, FTIR spectroscopy was considered as a qualitative tool but, since last two decades tremendous quantitative work has been published. Non-destructive and fast analytical nature of FT-IR spectroscopy can be exploited in an advantageous manner in wide range of application in modern industries [1]. Whereas diffuse reflectance infrared Fourier transform (DRIFT) is relatively better technique for quantitative analysis of solid state sample [2].

Aceclofenac is 2-[2-[2-[(2, 6-dichlorophenyl) amino] phenyl] acetyl] oxy acetic acid. It is a nonsteroidal anti-inflammatory drug with good analgesic effect [4] (Figure 1(A)). Pregabalin is S-3-(amino methyl)-5-methylhexanoic acid. It is an anticonvulsant drug for neuropathic pain and adjunct for partial seizures. It can be used in generalised anxiety disorders [5] (Figure 1(B)). ACF is official in British Pharmacopoeia, 2009, Indian Pharmacopoeia, 2007, 2010, and European Pharmacopoeia, 2005 [3 and 11]. PGL is official in Indian Pharmacopoeia, 2010 [3]. ACE is a non-steroidal anti-inflammatory drug which has analgesic and anti-inflammatory activity. ACE is an analog of diclofenac and it is a potent cyclooxygenase-2 inhibitor. ACE is official in IP, BP and USP. The pain management is always a trouble for a physician and searching for a safe and effective alternative is still on. The combination of the PGL (75 mg) and ACE (200 mg) is used for the relief of pain and inflammation in rheumatoid arthritis, osteoarthritis and ankylosing spondylitis. This combination is also used as an adjunctive in the treatment of partial seizures, epilepsy, fibromyalgia and neuropathic pain[13]. It is official in British Pharmacopoeia. The literature survey revealed that few analytical methods have been published concerning the simultaneous estimation of ACF and PGL either alone or in combination with other drugs, namely, UV spectrophotometric [4, 5, 6, 7] and chromatographic [8,9,10,11,12] methods. Several analytical techniques like titrimetric, colourimetric, spectrofluorimetric, densitometric, HPLC, RP-HPLC, spectrophotometric and stripping voltametric have been reported for assay of Aceclofenac. However some of these methods are costlier and time consuming. To overcome these difficulties spectrophotometric analysis serves to be the quickest, promising and reliable method for routine analytical needs[14].

Pregabalin (Lyrica TM) is an antiepileptic drug approved for a number of indications in the US and Europe that include adjunctive therapy of partial seizures in adults, pain from diabetic neuropathy or post-herpetic neuralgia in adults, and the treatment of anxiety disorders. Pregabalin chemically known as (3S)-3-(aminomethyl)-5-methylhexanoic acid is an antiepileptic, anticonvulsant and neurotransmitter. This drug produces its actions by binding to the alpha2-delta ( $\alpha 2\delta$ ) subunit of the voltage-gated calcium channels. More recently pregabalin has been approved by the FDA for the treatment of spinal cord injury and as the first drug indicated for the treatment of fibromyalgia and pregabalin received U.S.FDA approval for use in treating epilepsy, diabetic neuropathy pain and post herpetic neuralgia[15,16]. Recently, capillary electrophoresis and nuclear magnetic resonance technique was reported for PGL involving complexation with cyclodextrins [17]. The

chromatographic methods require high cost solvents in addition to tedious pretreatment, Regarding spectrophotometric methods for determination of PG; some of them don't offer high sensitivity or need tedious extraction procedures[18.]

According to literature survey, there was not any developed analytical method which has been reported for simultaneous estimation of PGL and ACF in combined dosage form. So an attempt was being made to a developed simple, accurate, precise, economical and reproducible FT-IR spectroscopic method for simultaneous estimation of PGL and ACF in combined tablet dosage form.

## **2. Experimental Work**

### **2.1. Reagents and Materials:**

ACF and PGL pure API were procured as a gift sample from Lupin Pharmaceuticals, Pvt. Ltd. and Concept Pharmaceutical Pvt. Ltd. Aurangabad. All employed chemicals were of Spectroscopic grade.

### **2.2. Instruments:**

Spectrophotometric measurements were carried on FT-IR spectrophotometer Serial no-A213747 (IR-Affinity-1, Shimadzu Corp., Japan) equipped with diffuse reflectance sampling interface and attached with computer operated Shimadzu IR solution software was used for collection and analyze the data. It also equipped with detector- DLATGS and uses a high-energy long life ceramic light source. FT-IR spectrum was recorded in range of 400-4000  $\text{cm}^{-1}$  with 45 scans and resolution of 8  $\text{cm}^{-1}$ . Analytical weighing balance (AA-2200), [Max.200g, Min. 0.01g; e = 0.0001g] and hot air oven were used during the study.

### **2.3 Diluent selection:**

Both the drugs were found to be compatible with potassium bromide. Here solid sampling method is used for FT-IR method development and validation. KBr is selected as diluent because it is transparent to IR- radiation and its peaks does not interfere with peaks of drugs

### **2.4 Preparation of working standard (0.5 % w/w):**

Accurately ACF (5mg) and PGL (5mg) pure drugs were mixed separately with 995 mg of KBr (spectroscopic grade). Triturated well and made homogenous mixture.

### **2.5 Selection of analytical wave number:**

Working standard (0.5 % w/w) of both drugs was scanned in IR range of 4000-400  $\text{cm}^{-1}$  with resolution of 4 and 45 scans. Wave number was selected in such a way that one functional group of one drug should not present in another drug so that one can avoid interference of one drug in another. Wave number (peak area) parameter is selected for both drugs, in that one can select wave number in a range. Functional group selected for ACF is  $-\text{NH}_2$  (amine) and wave number found in range of 3300-3200  $\text{cm}^{-1}$ . Functional group selected for PGL is  $-\text{COOH}$  (carboxylic acid) and wave number found in range of 1660-1600  $\text{cm}^{-1}$ . FT-IR spectrum of ACF, PGL and mixed standards are shown in Figure 2 and FT-IR spectrum of marketed tablet formulation, mixed standard and tablet excipients (Extracted from tablet formulation by using acetone sonicated for 30 min.) are shown in Figure 3.

## **2.6 Selection of analytical concentration range and linearity study (Preparation of calibration curve):**

Pure drug sample of ACF and PGL was diluted with KBr to get concentration ranging from 0.8-4.8 % w/w and 0.3-1.8 % w/w for ACF and PGL respectively. Peak area of these dilutions was measured in range of 3300-3200  $\text{cm}^{-1}$  and 1660-1600  $\text{cm}^{-1}$  for ACF and PGL respectively using KBr as blank. Plot of peak area versus concentration were found to be linear at respective wave number region and they are shown in Figure 4 and data are given in Table 1. The absorbance of ACF standard ( $\text{NH}_2$  group) in linearity spectrum and absorbance of PGL standard ( $-\text{COOH}$  group) in linearity spectrum are shown in Figure 5 and 6 respectively. The optical characteristic and other parameters are shown in Table 2.

## **2.7 Analysis of marketed tablet formulation:**

Twenty tablets of marketed formulation ACENAC-N were accurately weighed and average weight was calculated, then these tablets were crushed to fine powder. The quantity of powder equivalent to 40 mg of ACF and 15 mg of PGL were weighed and mixed with dried KBr, finely powdered KBr and dilution was made 4.0 % w/w for ACF and 1.5 % w/w for PGL respectively and scanned in in range of 4000-400  $\text{cm}^{-1}$  with resolution of 4  $\text{cm}^{-1}$  and 45 scans. The analysis procedure was repeated six times with tablet formulation. The results of analysis of marketed tablet formulation are given in Table 3.

## **3. Method Validation**

The proposed method was validated for accuracy, precision, and linearity, limits of detection (LOD) and quantification (LOQ), and reproducibility. The method validation was performed as per ICH Guidelines (ICH, Q2 (R1): 2005)

### **3.1 Linearity and range**

Calibration curves were prepared for six different ACF & PGL concentrations in the range of 0.8- 4.8 % w/w & 0.3-1.8 % w/w respectively. Required quantity of ACF was diluted with potassium bromide to get around 1000 mg and triturated to ensure sample homogeneity. Each calibration standard was analyzed in the replicates of six. Area under curve (AUC) corresponding to the amine peak around 3300-3200  $\text{cm}^{-1}$  & the carboxylic acid peak around 1660-1600  $\text{cm}^{-1}$  was used for the quantification and the average of six measurements was used to obtain the calibration curve for ACF and PGL respectively. The linearity of proposed method was found to be in between 0.8-4.8 % w/w for ACF and 0.3-1.8 % w/w for PGL. Regression coefficient is 0.995 & 0.995 for ACF and PGL respectively.

### **3.2 Precision**

The repeatability was evaluated by assaying six times the sample solution prepared for assay determination. Precision of the method was evaluated by inter day and intraday variation studies. In intraday studies, working solutions of standard and sample were analyzed thrice in a day and percentage relative standard deviation (% RSD) was calculated. In the inter day variation studies, working solution of standard and sample were analyzed on two consecutive days and percentage relative standard deviation (% RSD) was calculated. The result of repeatability study and intermediate precision study are given in Table 4 and Table 5 respectively.

### 3.3 Accuracy

To check the accuracy of the proposed method, recovery studies were carried out at 80%, 100% and 120% of the test concentration as per ICH guidelines. As per the label claim, tablet contains 200 mg of ACF and 75 mg of PGL. For recovery studies different levels of the standard concentration according to 80%, 100% and 120% are made and % mean recoveries was calculated. The result of recovery studies are given in Table 6 and graphical representation given in Figure 7.

### 3.4 Detection Limit and Quantitation Limit

ICH guideline describes several approaches to determine the detection and quantitation limits. These include visual evaluation, signal to noise ratio, and the use of standard deviation of the response and the slope of the calibration curve. In the present study, the LOD and LOQ were based on the third approach and were calculated according to the  $3.3\sigma/S$  and  $10\sigma/S$  criterions, respectively.

The LOD and LOQ may be calculated as;

$$LOD = 3.3 * \left( \frac{SD}{Slope} \right) \dots \dots \dots (1) \quad LOQ = 10 * \left( \frac{SD}{Slope} \right) \dots \dots \dots (2)$$

Where, SD is the standard deviation of Y-intercept of Calibration curves.

Slope is the mean slope of the calibration curves.

### 3.5 Determination of ACF and PGL in Their Combined Tablet Dosage Form

Accurately weighed 20 tablets of marketed formulation ACENAC-N and average weight were found to be 719 mg. This tablet were crushed to fine powder and from this powder equivalent to 200 mg of ACF and 75 mg of PGL was mixed with 143.8 drug powder mix with 856.2mg of KBr and dilution made to get 4 % w/w for ACF and 1.5 % w/w for PGL respectively.

### 3.6 Force Degradation Studies: (ICH, Q1A (R2); Q6A; Q5C; Q7; Q1B: 2005, Singh and Bakshi et al. 2000)

#### 3.6.1 Photolytic degradation:

Pure drugs were exposed to UV radiations for 6 hours and samples were withdrawn at interval of 30 min. The samples after exposure to light were diluted with KBr to get ACF(0.5% w/w) and PGL (0.5 % w/w). Peak area was measured at  $3300-3200\text{ cm}^{-1}$  &  $1660-1600\text{ cm}^{-1}$  for ACF and PGL respectively. Finally peak area of sample was compared with standard peak area and percent degradation and percent assay was calculated.

#### 3.6.2 Thermal degradation:

Thermal degradation was carried out by exposing pure drugs to dry heat at  $80^{\circ}\text{C}$  for hrs. Samples are withdrawn at interval of 30 min. The samples after exposure to heat diluted with KBr to get ACF (0.5 % w/w) and PGL (0.5 % w/w). Peak area was measured at  $3300-3200\text{ cm}^{-1}$  &  $1660-1600\text{ cm}^{-1}$  for ACF and PGL respectively. Finally peak area of sample was compared with standard peak area and percent degradation and percent assay was calculated.

#### 3.6.3 Degradation in Sunlight:

Sunlight degradation is performed by exposing the pure drugs to sunlight in open space for 6 hrs. Samples are withdrawn at interval of 30 min. The samples after exposure to sunlight diluted with KBr to get ACF (0.5 % w/w) and PGL (0.5 % w/w). Peak area was measured at 3300-3200  $\text{cm}^{-1}$  & 1600-1660  $\text{cm}^{-1}$  for ACF and PGL respectively. Finally peak area of sample was compared with standard peak area and percent degradation and percent assay was calculated

#### 4. Result and Discussion

In the present work, new IR spectrophotometric method was developed for determination of ACF and PGL. The linearity was observed in the concentration ranges between 0.8 – 4.8 %w/w for ACF and 0.3 – 1.8 %w/w for PGL with coefficients of correlation  $r^2=0.995$  and  $r^2=0.995$  for ACF and PGL at 3300-3200  $\text{cm}^{-1}$  & 16600-1660  $\text{cm}^{-1}$  respectively. Marketed formulation containing ACF and PGL was analysed by proposed method. Mean assay values in ACENAC-N tablet were found to be 99.84%±1.3794% RSD and 99.9997% ±0.2793 % RSD for ACF and PGL respectively. The accuracy of method was determined by recovery studies. Pure ACF and PGL were added to the pre analyzed tablet powder at three different levels i.e. 80%, 100% & 120% of label claims as per the ICH guidelines. The RSD value below 2% indicated that the method has required precision. The limit of quantitation of ACF and PGL were found to be 1.5504 %w/w and 0.8680%w/w respectively. Limit of detection was found to be 0.5116 %w/w and 0.2864 %w/w for ACF and PGL respectively. The proposed spectrophotometric method was successfully applied to ACF and PGL in combined tablet dosage form.

The stress degradation studies showed that ACF & PGL undergoes degradation thermal, photolytic and sunlight condition and percentage degradation was found to be 45.38, 42.66, 21.52 and 24.37, 42.12, 43.42 for ACF and PGL

A simple, rapid, accurate, precise, selective and reproducible FT-IR method for the quantitation of aceclofenac and pregabalin tablet was developed and validated. The results indicate the method to be sensitive, selective, accurate and reproducible. Sample preparation is very simple and it involves mixing of API and KBr. From the results of all the validation parameters we can conclude that the present method can be useful for simultaneous estimation of Aceclofenac and Pregabalin Tablets with desired precision and accuracy. The linearity range was from 0.8- 4.8 % w/w for aceclofenac and 0.3-1.8 % w/w for pregabalin. The developed method it will be validate as per ICH guidelines. The proposed method shows good agreement with all validation parameters. There should not be any interference from KBr and blank with main peak.

#### 5. Conclusion

The proposed FT-IR method was found to be simple, sensitive, accurate, and precise for estimation of ACF and PGL in combined dosage form. The common excipients and additives which are usually present in the combined dosage form do not interfere in the analysis of ACF and PGL in the method; hence it can be conveniently adopted for routine quality control analysis of the drugs in combined dosage form.

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**TABLE 1: Standard calibration data for ACF and PGL**

ACF		PGL	
Conc. (%w/w)	Peak area (3300-3200 cm <sup>-1</sup> )	Conc. (%w/w)	Peak area (1660-1600 cm <sup>-1</sup> )
0.8	32.718	0.3	11.347
1.6	38.835	0.6	15.078
2.4	43.436	0.9	17.938
3.2	47.284	1.2	20.777
4.0	54.086	1.5	25.048
4.8	58.750	1.8	28.875

**TABLE 2: Optical characteristics and other parameters**

Parameters	ACF	PGL
Wave number (cm <sup>-1</sup> )	3300-3200 cm <sup>-1</sup>	1660-1600 cm <sup>-1</sup>
Linearity range (%w/w)	0.8-4.8	0.3-1.8
y = mx ± c	y = 6.42x + 27.875	y = 11.466 + 7.804
Slope (m)	6.42	11.466
Intercept (c)	27.875	7.804
Regression coefficient (R <sup>2</sup> )	0.995	0.995
Limit of detection (% w/w)	0.5116	0.2864
Limit of quantitation (% w/w)	1.5504	0.8680

**TABLE 3: Assay of marketed formulation: ACENAC-N (ACF-200/PGL75)**

Drug	Label claim (mg)	Amount found (mg)*	% Found*	SD	% RSD
ACF	200	200.141	99.84	1.3772	1.3794
PGL	75	75.013	99.99	0.2793	0.2793

\*Indicates average of six determinations

**TABLE 4: Repeatability data of standard drug mixture**

Drug	% Found*	SD	% RSD
ACF	98.20	0.8961	0.9125
PRG	99.019	0.3836	0.3874

\*Indicates average of six determinations

**TABLE 5: Data for intra-day and inter-day precision**

Drug	Intra-day precision			Inter-day precision		
	% Found*	SD	% RSD	% Found*	SD	% RSD

ACF	99.893	0.1385	0.1386	99.038	1.6250	1.6407
PRG	99.84	0.0737	0.0739	98.44	1.3860	1.4079

\*Indicates average of six determinations

TABLE 6: Results of recovery study

Drug	Amount present	Amount added	Amount found*	% Recovery*	SD	%RSD
ACF	4	3.2	7.2	99.408	0.8501	0.8551
	4	4	8.0	99.689	0.4086	0.4099
	4	4.8	8.8	99.678	0.3494	0.3506
PGL	1.5	1.2	2.7	99.513	0.2442	0.2454
	1.5	1.5	3	99.564	0.3031	0.3044
	1.5	1.8	3.3	99.390	0.5069	0.5101

\*Indicates average of six determinations

TABLE 7: Data of forced degradation study

Degradation condition	% Degradation		% Assay	
	ACF	PGL	ACF	PGL
Thermal	45.38	24.37	54.62	75.63
Photolytic	42.66	42.12	57.34	57.88
Sunlight	21.52	43.42	78.48	56.58

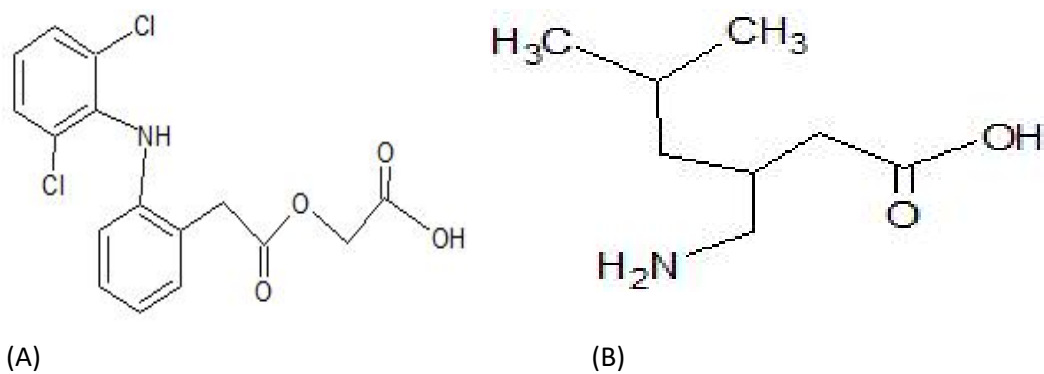
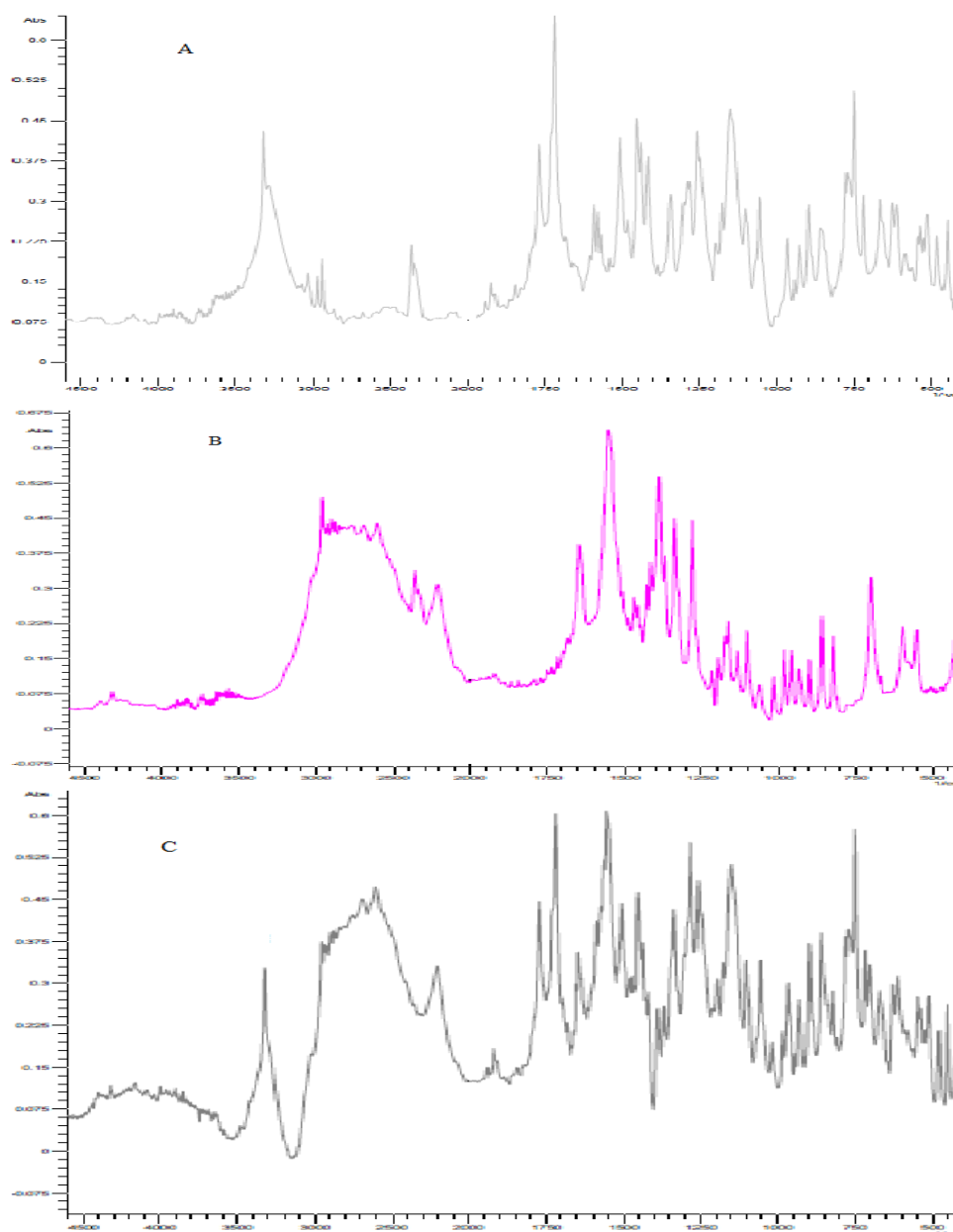
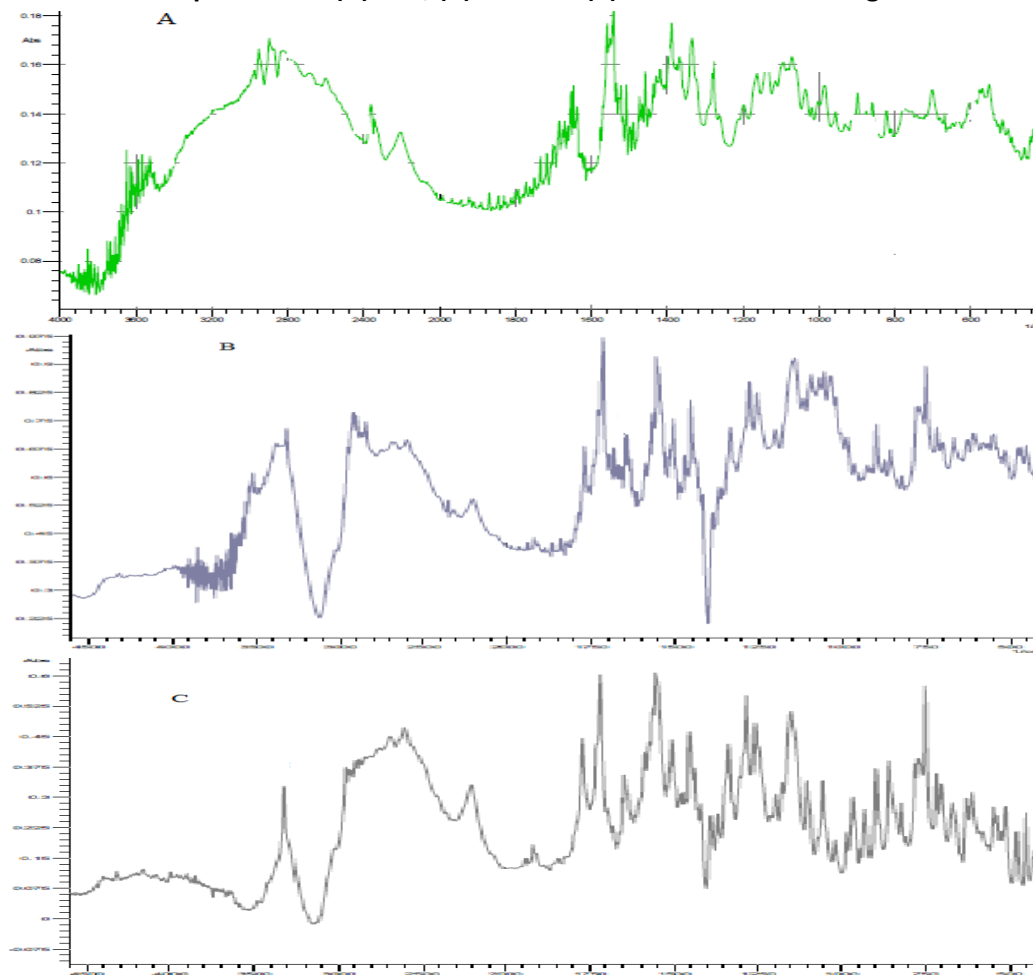


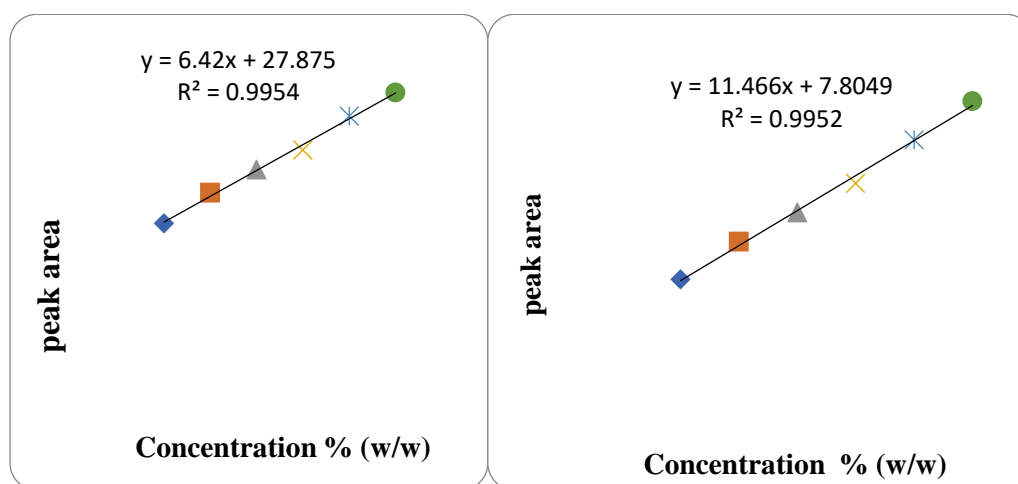
FIGURE 1: Chemical structure of (A) Aceclofenac and (B) Pregabalin



**FIGURE 2: FT-IR spectrum of (A) ACF, (B) PGL and (C) Mixed standard drugs**

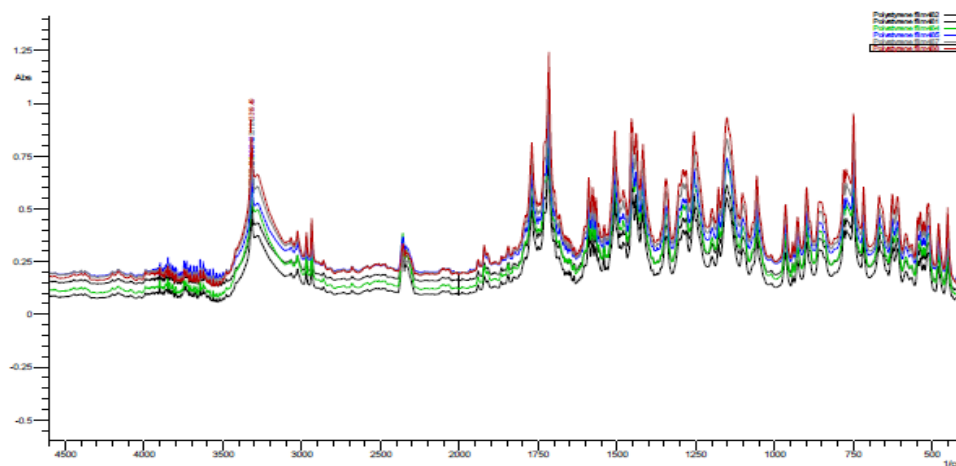


**FIGURE 3: FT-IR spectrum of (A) tablet excipients, (B) marketed tablet formulation and (C) mixed standard drugs**

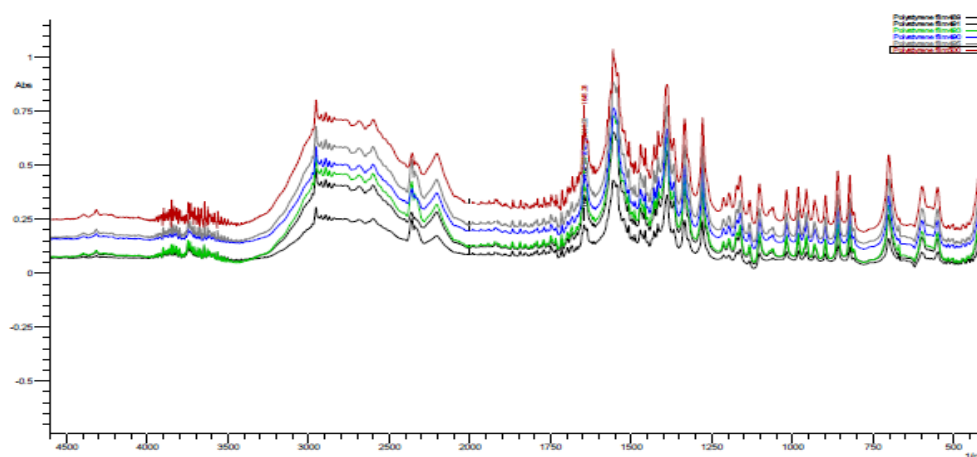


(a)(b)

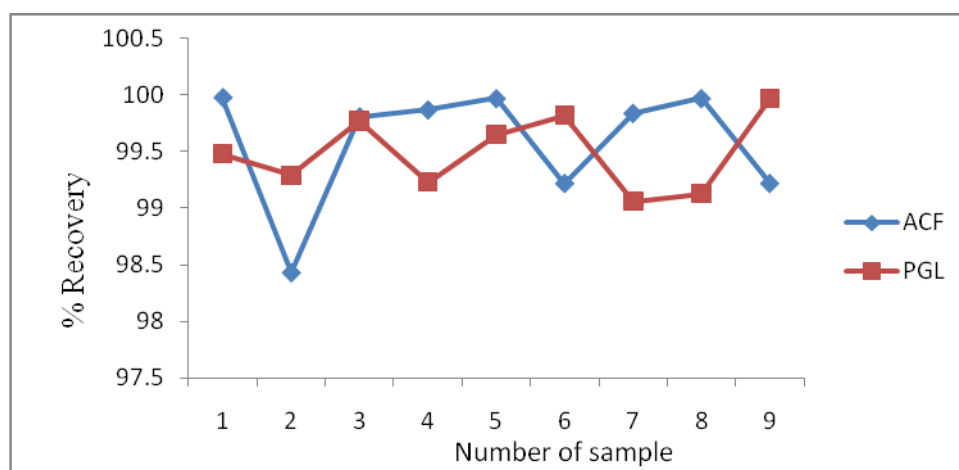
**FIGURE 4: Calibration curve for (a) ACF and (b) PGL**



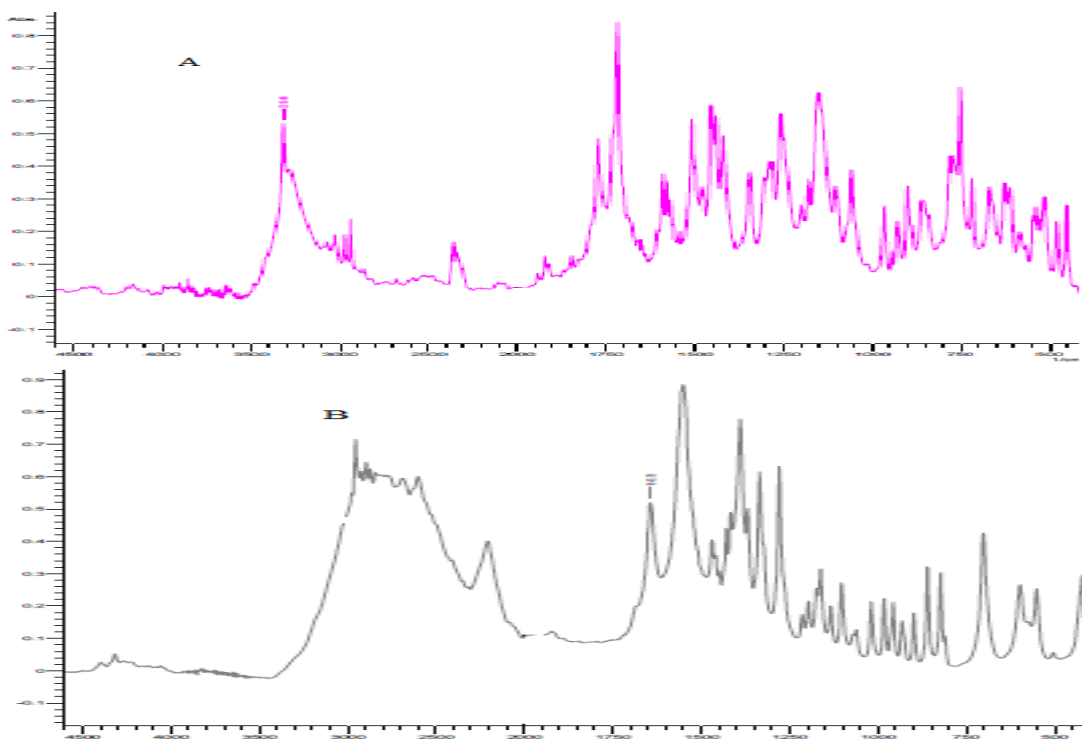
**FIGURE 5: Absorbance of ACF standard (NH<sub>2</sub>group) in linearity spectrum**



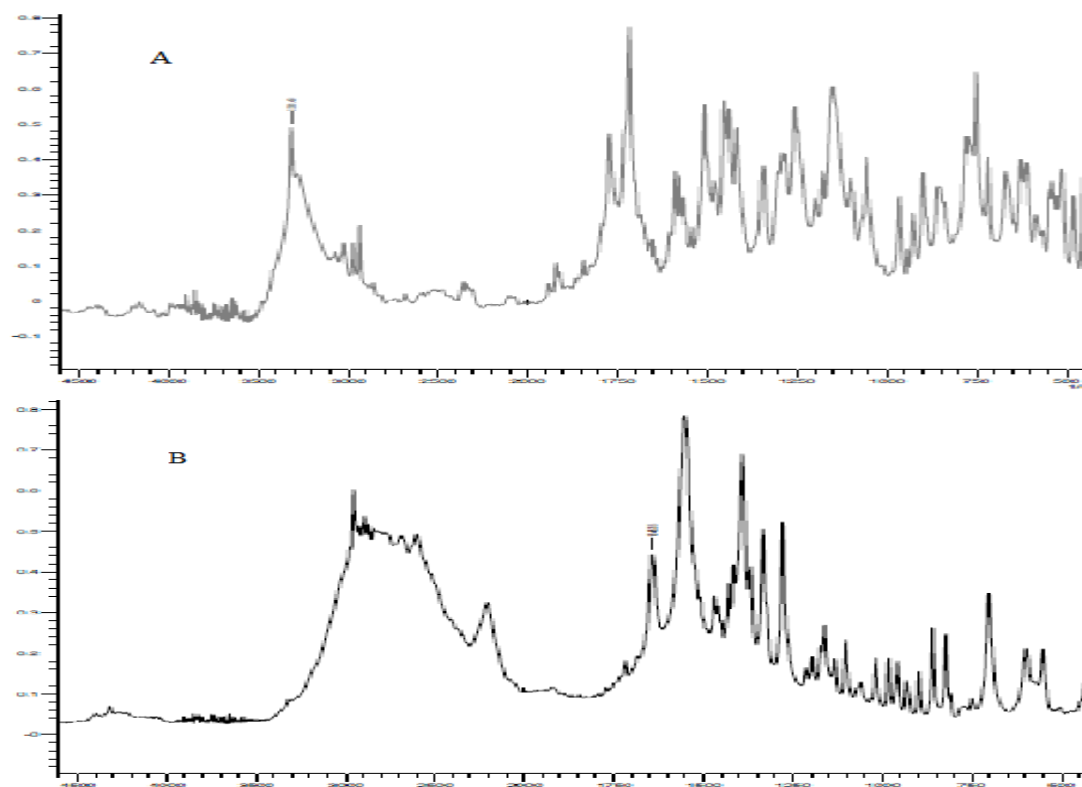
**FIGURE 6: Absorbance of PGL standard (-COOH group) in linearity spectrum**



**FIGURE 7: Graphical representation of result recovery study**



**FIGURE: 8** Thermal degradation of (A) ACF in 60 min. and (B) PGL in 30 min.



**FIGURE 9:** Photolytic degradation of (A) ACF in 90 min. and (B) PGL in 60 min.

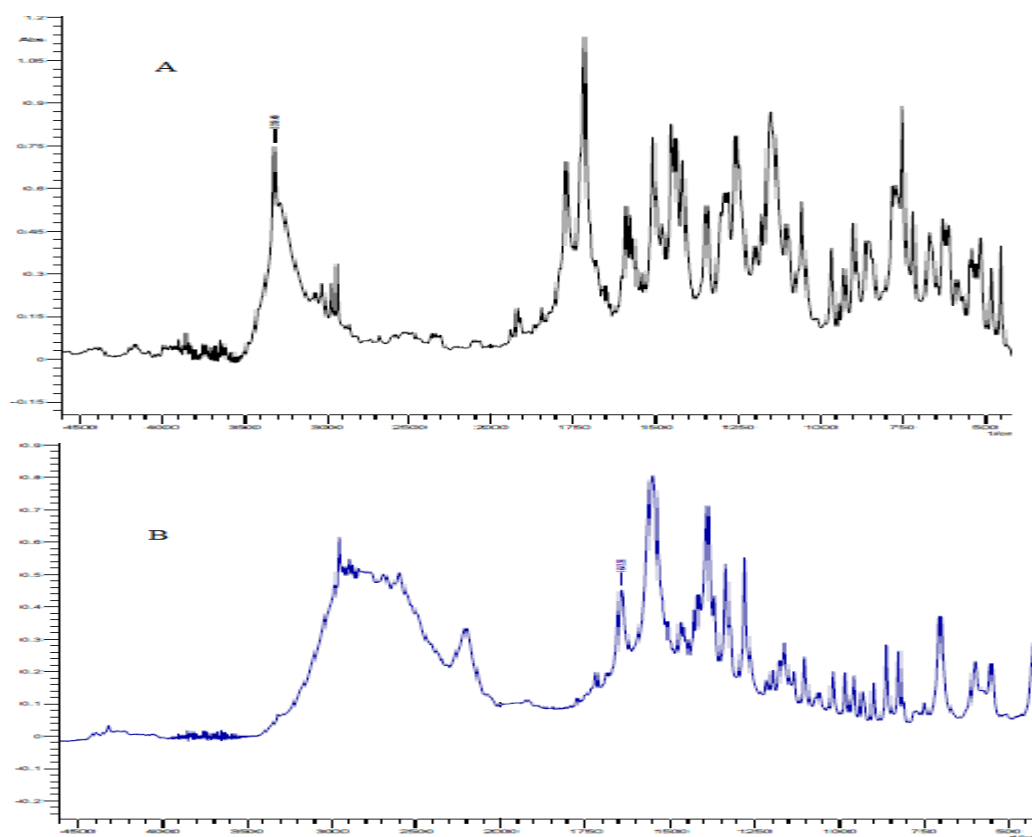


FIGURE: 10 Sun light degradation of (A) ACF in 60 min and (B) PGL in 60 min.