Naveen Kumar Reddy et al., Pharm Anal Acta 2011, 2:9
ÜÀí 10.4172/2153-2435.1000142

Pharmaceutica Analytica Acta

Research Article Open Access

Development and Validation of a Stability Indicating UPLC Method for Determination of Moxifloxacin Hydrochloride in Pharmaceutical Formulations

G. Naveen Kumar Reddy[1]*, V.V.S.Rajendra Prasad[2], Nigam Jyoti Maiti[3], Diptimayee Nayak[3] and Prashant Kumar Maharana[4]

[1]*Singhania University, Pacheri Bari, Jhunjhunu, Rajasthan*
[2]*Sitha Institute of Pharmaceutical Sciences, JNTU, Hyderabad*
[3]*Department of Pharmacy, IMT College of Pharmacy, Puri, Odisha*
[4]*Mannequin Pharmaceutical, Bhubaneshwar, Odisha*

Abstract

Simple, rapid, sensitive, accurate, robust & rugged stability indicating analytical method for determination of Moxifloxacin HCl in pharmaceutical formulations is developed and validated by using UPLC & applied the developed and validated method for determining the assay of Moxifloxacin HCl in tablets (Avelox®), as there is no official monograph & no analytical method by UPLC. Chromatography was performed with mobile phase containing potassium dihydrogen ortho phosphate (adjusted to pH 1.8 with orthophosphoric acid), Methanol & acetonitrile in the ratio of 60:20:20, with a flow rate of 0.3mL/min, C-18 column & UV detection at 296nm.The method was validated for linearity, accuracy, ruggedness, robustness, precision & bench top stability of sample & standard solution. Moxifloxacin tablets were subjected to different stress conditions like acid, alkali, peroxide, thermal, water & UV studies and checked for its specificity, degradation & stability. The developed method was very rapid with a run time of 3 min, accurate, robust, rugged and stable.

Keywords: Moxifloxacin; Assay method; UPLC; Stability indicating method

Introduction

Ultra performance liquid chromatography (UPLC) takes advantage of technological strides made in particle chemistry performance, system optimization, detector design, and data processing and control. Using sub-2 mm particles and mobile phases at high linear velocities, and instrumentation that operates at higher pressures than those used in HPLC, dramatic increases in resolution, sensitivity, and speed of analysis can be obtained. This new category of analytical separation science retains the practicality and principles of HPLC while creating a step function improvement in chromatographic performance [1].

According to an FDA guidance document, a stability-indicating method is "a validated quantitative analytical procedure that can detect the changes with time in the pertinent properties of the drug substance and drug product. A stability-indicating method accurately measures the active ingredients, without interference from degradation products, process impurities, excipients, or other potential impurities" [2].

Moxifloxacin is slightly yellow crystalline mono-hydrochloride salt [3]. Moxifloxacin Hydrochloride is designated chemically as ((1'S,6'S)-1-Cyclopropyl-7-(2,8-diazabicyclo[4.3.0]non-8-yl)-6-fluoro-8-methoxy-4-oxo-1,4-dihydroquinoline-3-carboxylic acid hydrochloride (Figure 1) [4]. Moxifloxacin can be used to treat respiratory infections, including acute sinusitis, acute exacerbations of chronic bronchitis, and community-acquired pneumonia, as well as skin and skin structure infections. Moxifloxacin is also used for the treatment of complicated intra-abdominal infections [5]. Moxifloxacin inhibits bacterial topoisomerase II (DNA gyrase) and topoisomerase IV. Topoisomerases are essential enzymes which play a crucial role in the replication and repair of bacterial DNA. This mechanism is lethal to susceptible bacteria. Moxifloxacin is often referred to as a chemotherapeutic drug because its mode of action has so far not been noted in any naturally occurring or semi-synthetic antibiotic.

A few methods for the determination of Moxifloxacin Hydrochloride in pharmaceutical formulations by HPLC [6], HPTLC [3] and UV [7] appear in literature. So far no systematic UPLC method has been reported for determination of Moxifloxacin Hydrochloride in pharmaceutical formulations. This paper reports a rapid and sensitive UPLC method with UV detection, useful for routine quality control of Moxifloxacin Hydrochloride in pharmaceutical formulations. The method was validated by parameters such as linearity, accuracy, precision, robustness, ruggedness, sample and standard solution stability and forced degradation studies.

Experimental

Reagents

HPLC grade Acetonitrile (HPLC Grade, Merck), Potassium dihydrogen orthophosphate (AR, Rankem), Hydrochloric Acid (AR, Rankem) Sodium hydroxide (AR, Rankem), Hydrogen peroxide (AR, Rankem), Ortho phosphoric acid (AR,Rankem),Water (Milli Q water), Acetonitrile (HPLC Grade, Merck). Moxifloxacin pure drug substance was kindly supplied by MSN Laboratories Limited, India. Ingredients used for placebo were microcrystalline cellulose, croscarmellose sodium, PVPK-30, Ethanol, Magnesium stearate.

***Corresponding author:** G. Naveen Kumar Reddy, Singhania University, Pacheri Bari, Jhunjhunu, Rajasthan, Tel: +91996340443; E-mail: naving29@gmail.com

Received November 03, 2011; **Accepted** November 18, 2011; **Published** November 21, 2011

Citation: Naveen Kumar Reddy G, Rajendra Prasad VVS, Maiti NJ, Nayak D, Prashant Kumar M (2011) Development and Validation of a Stability Indicating UPLC Method for Determination of Moxifloxacin Hydrochloride in Pharmaceutical Formulations. Pharm Anal Acta 2:142. doi:10.4172/2153-2435.1000142

Instrumentation

A liquid chromatography (Waters Acquity) system equipped with an injection valve (Rheodyne) & PDA detector. The UPLC system was well equipped with Empower 2 software for data processing. Other instruments like Sartorius Analytical Balance, Metrohm pH Meter and Biotechnics sonicator were used in sample and standard preparations

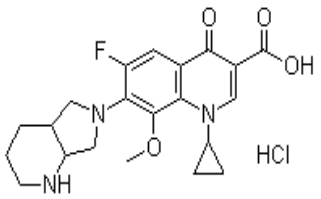

Figure 1: Moxifloxacin Hydrochloride.

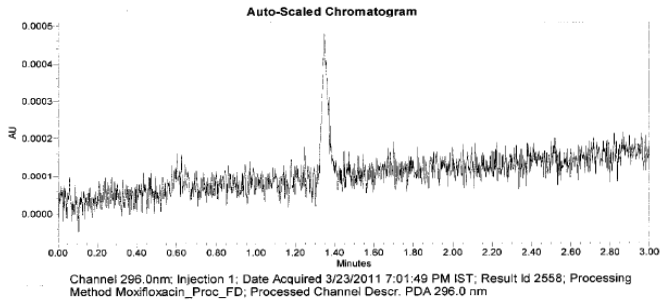

Figure 2: Blank-Diluent.

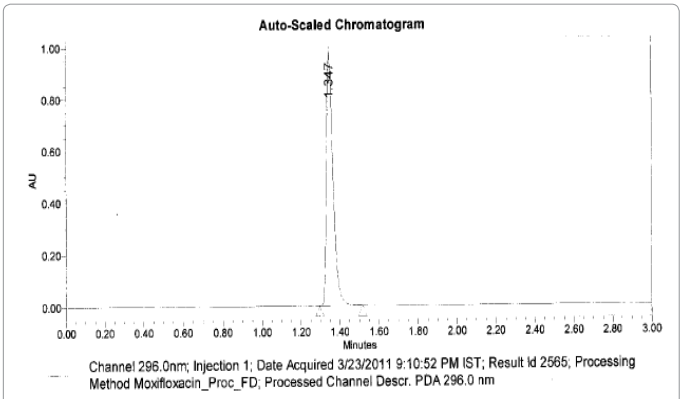

Figure 3: Standard.

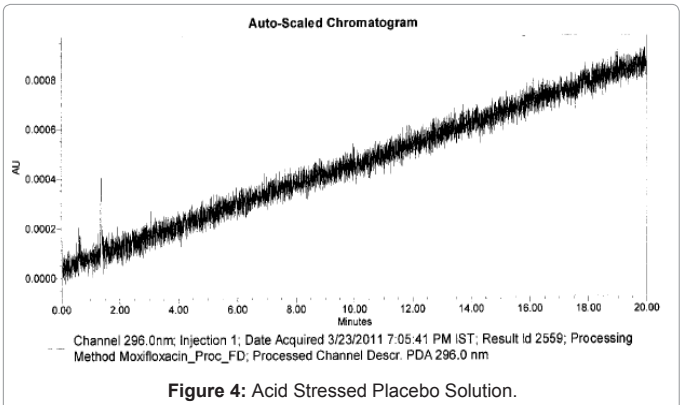

Figure 4: Acid Stressed Placebo Solution.

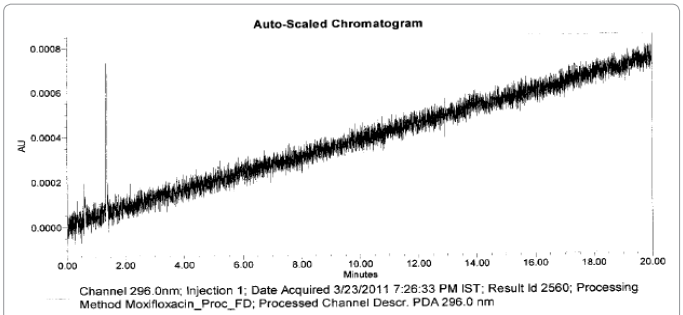

Figure 5: Alkali Stressed Placebo.

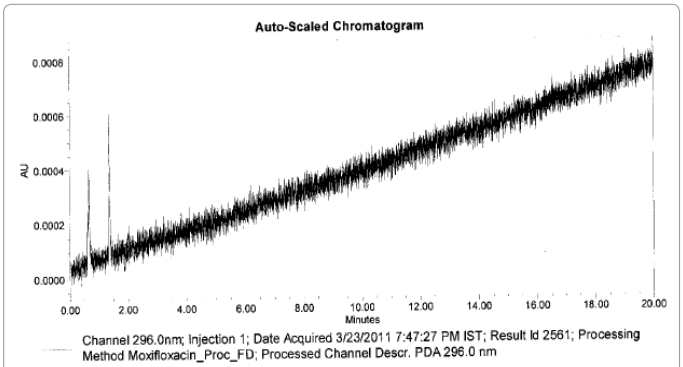

Figure 6: Peroxide Stressed Placebo.

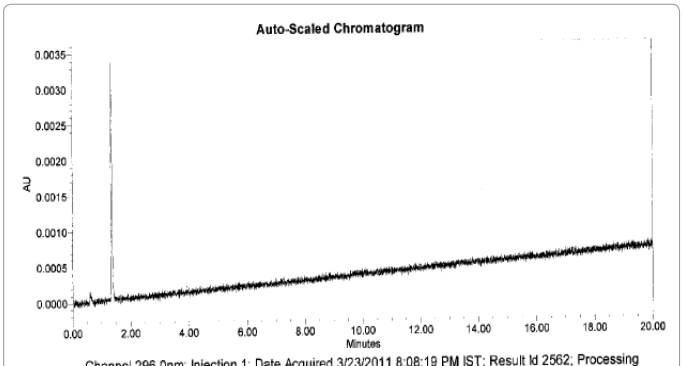

Figure 7: Water Stressed Placebo.

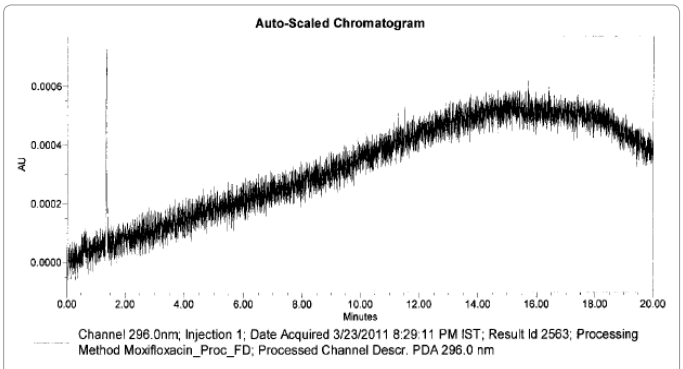

Figure 8: Heat Stressed Placebo.

and for forced degradation studies.

Methodology

Chromatographic conditions

The analytical column used was Waters HSS, C-18, 100X2.1; 1.8µm .The mobile phase was potassium dihydrogen ortho phosphate, adjusted to pH 1.8 with ortho phosphoric acid, methanol & acetonitrile in the ratio of 60:20:20. It has a flow rate of 0.3mL/min, injection volume of 1µL with ambient column oven temperature and sample tray temperature with isocratic elution & UV detection at 296nm & a run time of 3 min.

Standard, sample, mobile phase and diluent preparation

Diluent: Mobile phase is used as diluent:

Preparation of mobile phase: Dissolved 3.4g of potassium dihydrogen ortho phosphate in one litre water and adjusted the pH to 1.8 with ortho phosphoric acid.Filtered through 0.22µ membrane filter. Mixed the buffer,acetonitrile and methanol in the ratio of 60:20:20 and sonicated to degas.

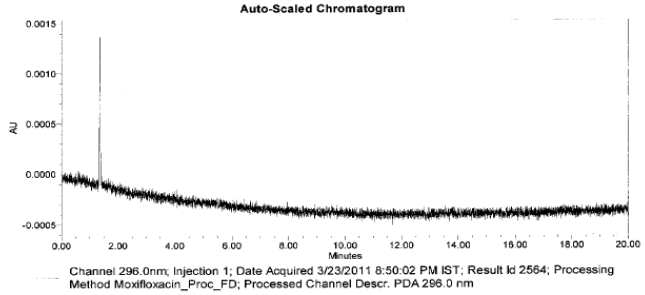

Figure 9: UV Stressed Placebo.

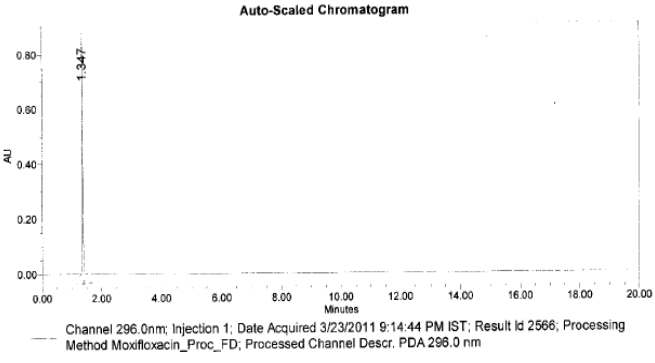

Figure 10: Acid Stressed Sample.

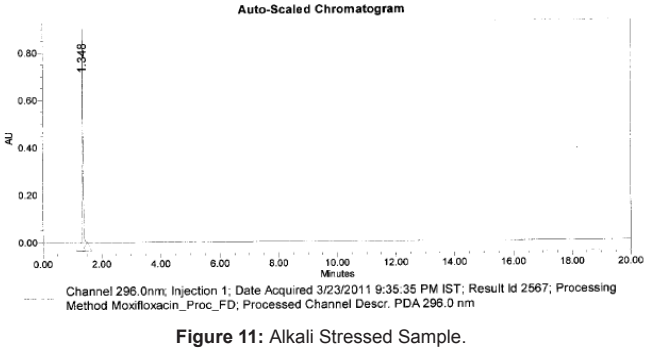

Figure 11: Alkali Stressed Sample.

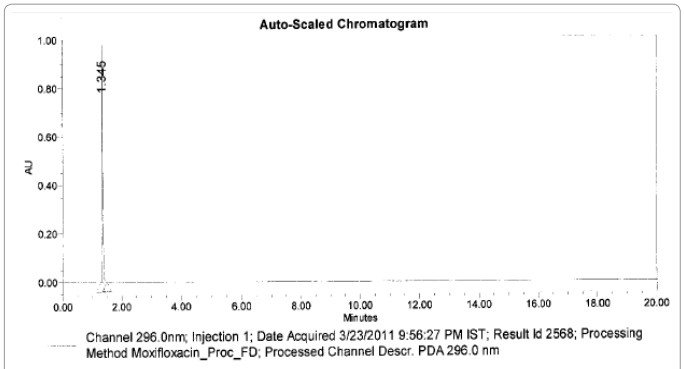

Figure 12: Peroxide Stressed Sample.

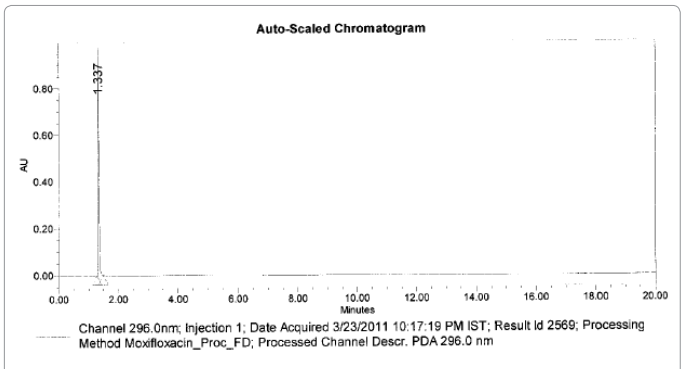

Figure 13: Water Stressed Sample.

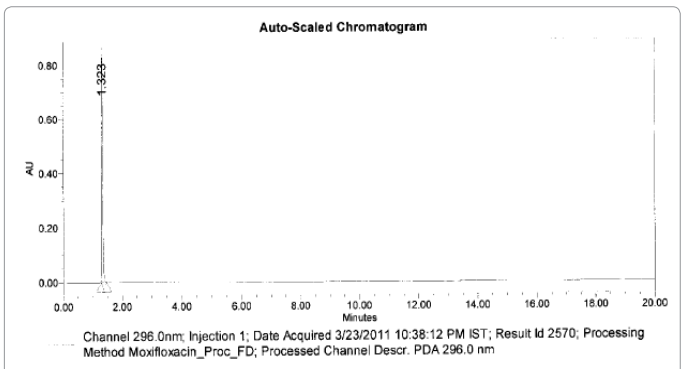

Figure 14: Heat Stressed Sample.

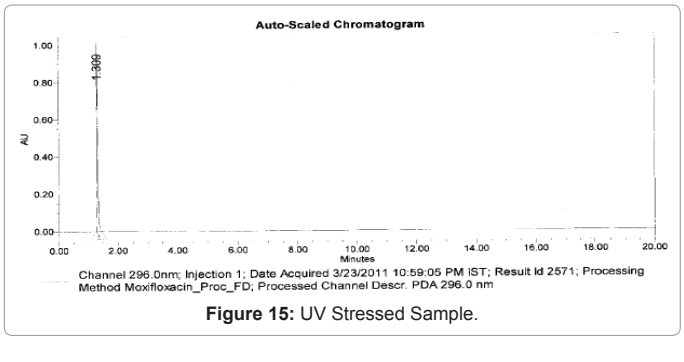

Figure 15: UV Stressed Sample.

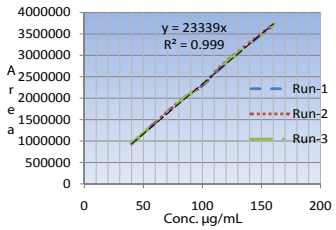

Figure 16: Linearity.

Preparation of standard solution: Accurately weighed and transferred 44mg of Moxifloxacin HCl in to a 100mL volumetric flask and added 70mL of diluent.Sonicated for 5 min and made up to the mark with diluent.Transferred 5mL of above solution to 20mL volumetric flask and made up to volume with diluent. Filtered with 0.45µm PFTE filter.

Preparation of Test solution: Weighed 20 tablets(Avelox-400mg) manufactured by Bayer Health Care AG, Germany and determined the average weight.Weighed 2 tablets and transferred in to a 200mL volumetric flask and added 150mL of diluent.Sonicated in cold water for 20minutes with intermittent shaking.Allowed it to cool to room temperature and diluted to volume with diluent. Filtered atleast 12mL of the above solution with 0.45µm PTFE filter and transfered 5mL of filtered solution to 200mL volumetric flask and made up to volume with diluent.

Method development

By selecting the HPLC method conditions from literature and by using the UPLC method convertor calculated the chromatographic conditions.

Wavelength was selected at 296nm based on the literature [6] and by scanning with PDA detector.

pH of the buffer was selected based on its pKa value.

Taken 0.05M Potassium di hydrogen phosphate and adjusted the pH to1.8±0.05 with OPA. By using buffer and ACN: MeOH (600:400) and by using the gradient programmes mentioned as in (Table 1) with HSS C-18,100X2.1,1.8µm column, flow rate of 0.3mL/min, injection volume(5µl),column oven temperature at 25°C injected the Moxifloxacin HCl standard.

In Trial -1 a split peak was observed at a retention time 34min, which might be because of more buffer. So changed the gradient programme with less buffer and more organic solvents as in Trial -2, in this case the peak was little broad and the retention time decreased to 7min.Then decreased the buffer as mentioned in Trial-3 and with that gradient programme and with an injection volume of 1µL injected Moxifloxacin HCl standard. In this it eluted at 3.2RT and the peak shape was good.

By considering all the aspects went for an isocratic elution with Buffer : (ACN: MeOH) 600:400 and with the above mentioned chromatographic conditions injected standard and test solutions. Peak shape, theoretical plates, RSD and tailing all were fine and within the limits.

Results and Discussion

Specificity

Specificity is the ability to assess unequivocally the analyte in the presence of components which may be expected to be present. Typically these might include impurities, degradants, matrix, etc. [8]. Specificity was demonstrated by injecting a blank, placebo and standard solution. No interference was seen at the retention time of analyte. The specificity was also demonstrated by induced degradation of Moxifloxacin formulation and placebo samples to acid degradation, alkali degradation, peroxide degradation, thermal degradation, water degradation, U.V. degradation. Purity angle is less than purity

Trial-1			**Trial-2**			**Trial-3**		
Time	Buffer % A	ACN:MeOH % B	Time	Buffer % A	ACN:MeOH % B	Time	Buffer % A	ACN:MeOH % B
0.00	100	0	0.00	85	15	0.00	70	30
10.00	95	5	10.00	85	15	3.50	70	30
15.00	85	15	15.00	57	46	4.50	30	70
30.00	70	30	30.00	56	44	5.50	70	30
40.00	40	60	40.00	30	70	7.00	70	30
345.00	100	0	45.00	85	15			
50.00	100	0	50.00	85	15			

Table 1:

MOXIFLOXACIN FORCED DEGRADATION				
Stress Condition	**Reagent Used**	**Conc.**	**Purity Angle**	**Purity Threshold**
Acid Stress	HCl	0.1N	0.120	0.271
Alkali Stress	NaOH	0.1N	0.124	0.274
Peroxide Stress	H_2O_2	3%	0.138	0.297
Water Stress	Water		0.140	0.277
Heat Stress	Heater	60°C	0.118	0.272
U.V. Stress	Photolytic chamber	1 Week	0.170	0.278
Acceptance Criteria	**Peak Purity shall pass**			

Table 2:

threshold for all the stress conditions. The results are tabulated in (Table 2), (Figures 2-15) represents different stress conditions.

System suitability Testing

System suitability testing is used to verify that the reproducibility of the system is adequate for the analysis to be performed. System suitability is done by preparing and injecting the standard solution 5 times and calculating its RSD. Other parameters like tailing and theoretical plates should also be taken in to consideration. Results are tabulated in (Table 3).

Linearity

The linearity of an analytical procedure is its ability (within a given range) to obtain test results which are directly proportional to the concentration (amount) of analyte in the sample [8]. The linearity of the test method was performed by plotting a graph between concentration of the test solution on X-axis and response of the corresponding solutions on Y-axis from 40% to 160% of test concentration and calculated the correlation coefficient, it was found to be 0.999.The results are tabulated in (Table 4 & 5) and the graphs are represented as Figure 16.

Limit of detection (LOD) and limit of quantification (LOQ)

The detection limit of an individual analytical procedure is the lowest amount of analyte in a sample which can be detected but not necessarily quantitated as an exact value. The quantitation limit of an individual analytical procedure is the lowest amount of analyte in a sample which can be quantitatively determined with suitable precision and accuracy [8]. Calculated the LOD & LOQ, with the calculations obtained from evaluation of the calibration curve of the linearity. LOD and LOQ values are less than the minimum linearity concentration.

The calculations and results are tabulated in (Table 6).

Bench top stability of standard & test preparation

Performed the assay of Moxifloxacin as per the test method in duplicate and kept the standard and test solutions on the bench top for 48 Hrs. Injected at initial, 24 Hrs. and 48 Hrs. Calculated the difference between initial and bench top stability samples for % assay of Moxifloxacin for test solutions and similarity factor for standard solutions were found to be within limits. The results are tabulated in (Table 7).

Accuracy

The accuracy of an analytical procedure expresses the closeness of agreement between the value which is accepted either as a conventional true value or an accepted reference value and the value found [8]. Performed the accuracy of test method using Moxifloxacin placebo at 50%, 70%, 100%, 125%, 150% spike levels. The % assay at each spike level was found to be between 98.0-102.0% of the labeled amount. The results are tabulated in (Table 8 and 9).

Precision

The precision of an analytical procedure expresses the closeness of agreement (degree of scatter) between a series of measurements obtained from multiple sampling of the same homogeneous sample under the prescribed conditions. Precision may be considered at three levels: repeatability, intermediate precision and reproducibility [8].

Method precision

Determined the precision of the test method by preparing & injecting 6 test solutions of Moxifloxacin formulations in to the chromatograph and recorded the results. The average % assay was found to be 100.4 with % RSD of 0.62. The results are tabulated in (Table 10).

Intermediate precision

Performed the assay of Moxifloxacin by following the same procedure as that of Method precision but on a different day and by a different analyst. The average % assay was found to be 99.4% with % RSD of 0.39.Overall RSD when compared with Method precision is 0.73. The results are tabulated in (Table 11 and 12).

Moxifloxacin System Suitability									
Injection No.:	1	2	3	4	5	Mean	STDEV	RSD	***Limits***
Standard Area:	2305687	2302824	2311478	2300543	2283295	2300765	10589	0.5	**RSD NMT 2.0%**
Theoretical Plates	7818	7835	7825	7826	7829	7827	6.19	0.1	**NLT 2000**
USP tailing	1.54	1.54	1.54	1.54	1.53	1.54	0.00	0.3	***NMT 2.0***
RT	1.259	1.260	1.263	1.265	1.267	1.263	0.00	0.3	

Table 3 :

Moxifloxacin Weighed	**Equivalent to mg**	**MOXIFLOXACIN-LINEARITY**			
		Diluted to(mL)	**mL**	**mL**	**Conc. (µg/mL)**
43.64	40.02	100	2	20	40.02
43.64	40.02	100	4	20	80.04
43.64	40.02	100	5	20	100.04
43.64	40.02	100	6	20	120.05
43.64	40.02	100	8	20	160.07

Table 4 :

MOXIFLOXACIN-LINEARITY						
Run	**% Conc.**	**Conc. Of Moxifloxacin (µg/mL)**	**Area of Moxifloxacin**	**Slope**	**Y-intercept**	**R^2**
1	**40%**	**40.02**	**937722**	**23058.3**	**27292.95**	**0.999**
	80%	**80.04**	**1908256**			
	100%	**100.04**	**2295800**			
	120%	**120.05**	**2819056**			
	160%	**160.07**	**3709937**			
2	**40%**	**40.02**	**942173**	**23183.8**	**25535.15**	**0.999**
	80%	**80.04**	**1908189**			
	100%	**100.04**	**2301865**			
	120%	**120.05**	**2852614**			
	160%	**160.07**	**3719921**			
3	**40%**	**40.02**	**943469**	**23069.1**	**31258.15**	**0.999**
	80%	**80.04**	**1902911**			
	100%	**100.04**	**2306901**			
	120%	**120.05**	**2831549**			
	160%	**160.07**	**3711182**			
Average				**23103.74846**	**28028.75**	**0.999**
Standard Deviation				**69.55**	**2931.59**	**0.00**
Acceptance criteria: Coefficient of correlation shall be NLT 0.999						

Table 5:

Moxifloxacin- Limit of detection (LOD) & Limit of Quantification (LOQ)				
S.No.	Injection No.	Slope	Y-Intercept	R^2
1	Inj-1	23059.4	27156.504	0.999
2	Inj-2	23184.9	25399.4381	0.999
3	Inj-3	23070.2	31121.98583	0.998
Average		23104.8333	27892.6426	0.9987
STDEV		69.550	2931.435	0.001
LOD=3.3 x σ/S				
LOD	0.4	ppm		
LOQ=10 x σ/S				
σ = Standard deviation of y-intercepts of regression line				
S= slope of the linearity curve				
LOQ	1.3	ppm		
Acceptance Criteria: LOD & LOQ values shall be less than the minimum linearity concentration				

Table 6:

Robustness

The robustness of an analytical procedure is a measure of its capacity to remain unaffected by small, but deliberate variations in method parameters and provides an indication of its reliability during normal usage [8]. Robustness was performed by injecting the Moxifloxacin standard solution in to the UPLC by altering the Flow rate, Column oven temperature and also by changing the pH of the buffer & composition of the organic solvent from the normal

Moxifloxacin Bench Top Stability of Standard Solution						
Time(Hrs)	**Day**	**Std. Wt.**	**Response**	**Fresh Std Wt.**	**Response of fresh std.**	**Similarity Factor**
Initial	Initial	44.02	2300765			
24 Hrs	Day-1	44.02	2311082	44.13	2316978	1
48 Hrs	Day-2	44.02	229288	43.89	2268919	0.99
Acceptance Limits: Similarity Factor should be NMT 2.0						
Moxifloxacin Bench Top Stability of Test Solution-1						
Time(Hrs)	**Day**	**Weight(mg)**	**Response of sample**	**% Assay**	**Difference from Initial**	**Difference in Assay results of Initial,24 & 48 Hrs shall be NMT 2.0**
Initial	Initial	1353.34	2337254	101.29	NA	
24 Hrs	Day-1	1353.34	2331881	100.6	0.7	
48 Hrs	Day-2	1353.34	2305445	101.01	0.3	
Moxifloxacin Bench Top Stability of Test Solution-2						
Time(Hrs)	**Day**	**Weight(mg)**	**Response of sample**	**% Assay**	**Difference from Initial**	**Difference in Assay results of Initial,24 & 48 Hrs shall be NMT 2.0**
Initial	Initial	1351.89	2321427	100.6	NA	
24 Hrs	Day-1	1351.89	2320794	100.12	0.5	
48 Hrs	Day-2	1351.89	2327728	101.99	1.4	

Table 7:

Standard Preparation	**44.13**	**mg**	**5**	**Potency**	**98.8**		
						Molecular factor of Moxifloxacin	
Sample Preparation	**Wt. of sample taken in mg**		**5**	**Label Claim**	**400**		**0.917**
	200		**200**				
Standard Area		**2316978**		**Average Wt. in mg**		**675.01**	

Table 8:

MOXIFLOXACIN-ACCURACY							
Spike level	**Wt.of sample taken in mg**	**Sample area**	**mg/mL added**	**mg/mL found**	**% Recovery**	**% Recovery**	**Average**
50%_01	674.46	1159290	0.04996	0.05454	100.1	100.1	
50%_02	672.90	1155954	0.04984	0.05438	100.1	100.0	100.1
50%_03	673.11	1158198	0.04986	0.05449	100.2	100.2	
70%_01	1018.65	1753515	0.07545	0.08249	100.3	100.3	
70%_02	1018.42	1746671	0.07544	0.08217	99.9	99.9	100.0
70%_03	1016.46	1744562	0.07529	0.08207	100.0	100.0	
100%_01	1349.09	2292178	0.09993	0.10783	98.9	99.0	
100%_02	1348.20	2281190	0.09987	0.10732	98.5	98.5	98.6
100%_03	1347.63	2272375	0.09982	0.10690	98.2	98.2	
125%_01	1686.17	2867979	0.1249	0.13492	99.1	99.1	
125%_02	1685.31	2856118	0.12484	0.13436	98.7	98.7	98.9
125%_03	1685.91	2866778	0.12488	0.13487	99.0	99.0	
150%_01	2015.68	3400552	0.14931	0.15998	98.3	98.3	
150%_02	2023.69	3406155	0.1499	0.16024	98.0	98.0	98.2
150%_03	2021.14	3411601	0.14971	0.16050	98.3	98.3	
Acceptance criteria:% Average recovery shall be between 98.0% -102.0%							

Table 9:

Moxifloxacin Analytical Method Validation-Assay								
Method Parameter			**Method Precision**					
Std. wt. & Dilution	**44.02**	**5**	**Tablet Wt.**	**Spl. wt. & Dilution**	**Wt. of sample taken**	**5**	**Label claim (mg)**	**400**
	100	**20**	**675.01**		**200**	**200**	**Potency (%)**	**98.8**
Molecular factor for Moxifloxacin				**0.917**				
Std. No.	**Standards**	**USP Tailing**	**Weight of sample taken**	**Area of sample**	**Assay %**	**Average (%)**	**STDEV**	**% RSD**
1	2310915	1.54	1353.34	2337254	101.04	100.4	0.61837	0.62
2	2290693	1.54	1351.89	2321427	100.46			
3	2300684	1.54	1358.15	2317128	99.81			
4	2300777	1.54	1353.97	2341249	101.16			
5	2300755	1.54	1355.02	2324067	100.34			
			1356.39	2310208	99.64			
Average	2300765	1.54	1354.79	2325222	100.41			
STDEV	7149.73	0.00	**Limits**	**% RSD of 6 replicate injections is not more than 2**				
%RSD	0.31	0.0						

Table 10:

Moxifloxacin Analytical Method Validation-Assay								
Method Parameter			**Intermediate Precision**					
Std. wt. & Dilution	**44.13**	**5**	**Tablet Wt.**	**Sample wt. & Dilution**	**Wt. of sample taken**	**5**	**Label claim (mg)**	**400**
	100	**20**	**675.01**		**200**	**200**	**Potency (%)**	**98.8**
Molecular factor for Moxifloxacin				**0.917**				
Std. No.	**Standards**	**USP Tailing**	**Wt. of sample taken**	**Area of sample**	**Assay %**	**Average (%)**	**STDEV**	**% RSD**
1	2315498	1.52	1351.91	2303175	99.22	99.4	0.388	0.39
2	2302693	1.52	1360.40	2318575	99.26			
3	2314434	1.52	1355.75	2314650	99.43			
4	2321577	1.52	1353.39	2305262	99.20			
5	2330688	1.52	1352.51	2325271	100.13			
6			1356.55	2306776	99.03			
Average	2316978	2	1355	2312285	99.38			
STDEV	10269.35	0.00	**Limits**	**% RSD of 6 replicate injections is not more than 2**				
%RSD	0.4	0.0						

Table 11:

Moxifloxacin Analytical Method Validation-Assay							
Method Parameter		**Method & Intermediate Precision combined**					
Method Precision		**Intermediate Precision**					
S.No.	**% Drug content**	**S.No.**	**% Drug content**	**Difference**	**Average of both Method & Intermediate precision**	**STDEV of both Method & Intermediate precision**	**%RSD of both Method & Intermediate precision**
1	101.04	1	99.2	1.8	99.9	0.730	0.73
2	100.46	2	99.3	1.2			
3	99.81	3	99.4	0.4			
4	101.16	4	99.2	2.0			
5	100.34	5	100.1	0.2			
6	99.64	6	99.0	0.6			
Limits: Overall RSD when compared with Method precision should be not more than 2%.							

Table 12:

Moxifloxacin Analytical Method Validation-Assay					
Method Parameter			**Robustness**		
Change in Flow Rate(0.25mL/min)			**Change in Flow Rate(0.35mL/min)**		
Std. No.	**Standards**	**USP Tailing**	**Std. No.**	**Standards**	**USP Tailing**
1	2743760	1.55	1	1973875	1.49
2	2774673	1.55	2	1943344	1.49
3	2740829	1.55	3	1960245	1.49
4	2732432	1.55	4	1952056	1.49
5	2734277	1.55	5	1958542	1.49
Average	2745194	1.55	**Average**	1957612	1.49
STDEV	17118.49	0.00	**STDEV**	11255.31	0.00
%RSD	0.62	0.0	**%RSD**	0.57	0.0
Change in pH of Mobile Phase(1.6)			**Change in pH of Mobile Phase(2.0)**		
Std. No.	**Standards**	**USP Tailing**	**Std. No.**	**Standards**	**USP Tailing**
1	2271424	1.49	1	2263481	1.53
2	2252217	1.49	2	2258739	1.53
3	2249439	1.49	3	2276006	1.53
4	2244184	1.49	4	2272593	1.53
5	2241573	1.48	5	2276184	1.53
Average	2251767	1.49	**Average**	2269401	1.53
STDEV	11762.64	0.00	**STDEV**	7882.71	0.00
%RSD	0.52	0.3	**%RSD**	0.35	0.0
Change in Org Phase Composition (90%)			**Change in Org Phase Composition (110%)**		
Std. No.	**Standards**	**USP Tailing**	**Std. No.**	**Standards**	**USP Tailing**
1	2311223	1.43	1	2265737	1.53
2	2313683	1.43	2	2269570	1.53
3	2305552	1.43	3	2290266	1.53
4	2315524	1.43	4	2291368	1.53
5	2306395	1.43	5	2290691	1.53
Average	2310475	1.43	**Average**	2281526	1.53
STDEV	4393.90	0.00	**STDEV**	12742.53	0.00
%RSD	0.19	0.0	**%RSD**	0.56	0.00

Table 12:

chromatographic conditions. The results are tabulated in (Table 13).

Calculation:

%Assay:

$$\frac{At}{As} \times \frac{Ws}{100} \times \frac{5}{20} \times \frac{200}{Wt} \times \frac{200}{5} \times \frac{P}{100} \times \frac{100}{L} \times 100 \times MF=$$

Where

At=Area of test solution; P=Potency of Moxifloxacin HCl Working Std.on as is basis

As=Area of standard solution; Avg. Wt. =Avg. Wt. of 20 tablets

Ws=Weight of standard taken; LC=Label claim of the tablet as Moxifloxacin

Wt=Weight of two tablets; MF=Molecular Factor for Moxifloxacin (0.917)

Conclusion

The reported UPLC method was proved to be simple, rapid with a runtime of 3 min & reproducible. The validation data indicates good specificity, precision, accuracy & reliability of the method. The developed method has many advantages like isocratic mode of elution, easy sample preparation, short run time and can be used for routine quality control analysis of Moxifloxacin formulations.

References

1. UPLC™: An Introduction and Review Michael E. Swartz Waters Corporation, Milford, Massachusetts, USA

2. FDA Guidance for Industry. Analytical Procedures and Methods Validation

(draft guidance), August 2000.

3. Vandana, Alok Kumar Chaudhary (2010) AJPSP 1:1, November 15, 74-84.
4. www.drugbank.ca/drugs/DB00218
5. www.chemblink.com
6. Kumudhavalli Manni Venkatachari,Chidambaram Saravanan, Rama Subbaiah, Balasundaram Jayakar (2010) IRJP 1(1) 333-336.
7. Dhumal DM, Shirkhedkar AA, Surana SJ (2011) Der Pharmacia letter 3: 453-456.
8. Validation of Analytical Procedures: Text And Methodology Q2(R1).