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Development and validation of stability indicating RP-HPLC method for Simultaneous determination of Desloratadine and Montelukast Sodium in pharmaceutical dosage form

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ABSTRACT

A simple, precise cost effective and stability indicating RP-HPLC method has been developed and validated for simultaneous determination of Desloratadine (DES) and Montelukast sodium (MKT) in pharmaceutical formulation. The RP-HPLC was performed on Zodiac C_{18}, (100mmx4.6mm, 5µ particle size) column. Mobile phase consisting of mixed buffer and methanol in the ratio 40:60 v/v. (2.6 gms of potassium hydrogen phosphate and 0.6 gms of dipotasssium hydrogen phosphate in 1000 ml distilled water, pH was adjusted to 6.0±0.1 with dilute orthophosphoric acid) and column temperature was 30°C. The flow rate of the mobile phase was 1.0 ml/min and the injection volume was 20µl. Detection was performed at 261nm.The Retention time for DES and MKT were 2.460 and 3.129 min respectively. The method was validated and shown to be linear for DES and MKT in 2.5-15.0 µgmL^{-1} and 5.0-30.0 µgmL^{-1} respectively. The regression was 0.9997 for DES and 0.9999 for MKT. DES and MKT were highly susceptible to acid (33.18 & 35.49), peroxide conditions (35 & 32.39) and least susceptible to UV (2.22 & 2.52) and basic conditions (2.59 & 3.73). Precision studies showed % RSD values less than 2% for both the drugs in all the selected concentrations. The percentage recoveries of MKT and DES were in the range of 99.40%-99.99%and 99.18%-100.45% respectively. The limit of detection (LOD) and limit of quantification (LOQ) were 0.084µg/ml, 0.254µg/ml for MKT and 0.081 µg/ml, 0.246µg/ml for DES respectively. The method was validated as per the International Conference on Harmonization (ICH) guidelines.

Keywords: Desloratadine, Montelukast Sodium, Stability Indicating Assay Method and RP-HPLC.

INTRODUCTION

Montelukast Sodium (MKT) (Figure 1) is chemically 2-[1-({[(1R) -1-{3-[(E) -2-(7-chloroquinolin-2-yl) ethenyl] phenyl} -3-[2-(2-hydroxypropan-2-yl) phenyl] propyl] sulfanyl} methyl) cyclopropyl] acetic acid [1]. It has a molecular formula of $C_{35}H_{36}ClNO_3S$ and molecular weight is 586.184g/Mol. MKT is a leukotriene receptor antagonist (LTRA) used for the maintenance treatment of asthma and to relieve symptoms of seasonal allergies [2] [3]. MKT selectively antagonizes leukotriene D4 (LTD4) at the cysteinyl leukotriene receptor, CysLT1, in the human airway. MKT inhibits the actions of LTD4 at the CysLT1 receptor, preventing airway edema,

smooth muscle contraction and enhanced secretion of thick, viscous mucus [4] -[7].

Desloratadine (DES) (Figure 2) is chemically 8-chloro-6, 11-dihydro-11-(4-deserdinylidene)-5*H*-benzo [5] [6]cyclohepta[1, 2-b]pyridine [3]. It has a molecular formula of $C_{19}H_{19}ClN_2$ and molecular weight is 310.82g/Mol. DES is a second generation, a tricyclic antihistamine that which has a selective and peripheral H1-antagonist action [4]. It is the active descarboethoxy metabolite of Loratidine. DES has a long-lasting effect and does not cause drowsiness because it does not readily enter the central nervous system [5] -[7].

The literature survey reveals that Montelukast sodium is estimated individually by UV spectrophotometry [8,9], spectrofluorometry [10], RP-HPLC [11,12], HPTLC [13,14], plasma HPLC [15,16,17,18], LC/MS [19,20], and stability indicating HPLC methods [21,22]. Similarly for Desloratadine the LC-ESI-MS/MS method was developed for quantitation of DES in human plasma and applied for its pharmacokinetic study [23] .Other methods for quantification of DES are in rat plasma with LC–MS[24], human plasma with LC–MS[25, 26,27,28] , LC–MS with nanospray ionization[29] and human plasma with HPLC [30,31,]. Another method was developed using liquid–liquid extraction [32] with a concentration range of 0.1–20 ng/ml. The sensitivity was improved with a concentration range of 0.05–10 ng/ml [33]. Furthermore, the spectrofluorometric method (Method II) was reported in the in vitro determination of DES in the presence of the parent drug loratadine in spiked human plasma and samples were prepared using solid phase extraction [34].

MKT with DES is used for the treatment of persistent allergic rhinitis [35]. The literature survey reveals that few analytical methods for the simultaneous estimation of MKT and DES in pharmaceutical formulations by using UV [36-38] and HPLC [39-42]. Hence, we made an attempt to develop a simple method for the simultaneous estimation of MKT and DES by RP-HPLC in pharmaceutical dosage forms. The proposed method was optimized and validated as per the International Conference on Harmonization (ICH) guidelines [43].

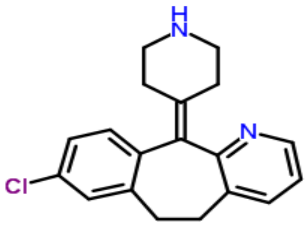


Fig: 1 Desloratadine

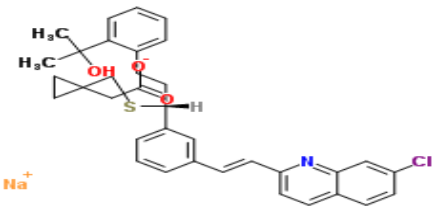


Fig: 2 Montelukast Sodium

Experimental

Materials and Methods Chemicals and Reagents

Dipotasssium hydrogen phosphate and potassium hydrogen phosphate and orthophosphoric acid were bought from SR Scientifics - Tirupati, India. Acetonitrile (HPLC grade) purchased from SR Scientifics - Tirupati, India. Bio Leo Labs pLtd. Hyderabad, Telangana, India was kind enough and supplied the reference standards of Desloratadine and Montelukast sodium for this research work. All the chemicals used throughout the research work were of analytical grade. Commercial tablets of Desloratadine (5 mg) and Montelukast sodium (10 mg) was purchased from local market manufactured by Bio Leo Labs pLtd. Hyderabad, India

Instrumentation

Waters HPLC 2 2695 series consisting pump, Auto sampler, UV-Vis detector, Thermostat column compartment, connected with Waters (alliance) Empower software.

In addition, an electronic balance (Shimadzu TX223L), digital pH meter (Systronics model 802), a sonicator (spectra lab, model UCB 40) was used in this present study.

Chromatographic conditions

The chromatographic separation was performed on Zodiac C18, 100x4.6, 5µ. The column temperature was kept at 30ºC. Separations were performed in isocratic mode using a mobile phase consisting of mixed buffer and methanol in the ratio of 40: 60. (P^H6.0 ±0.1 adjusted by using Ortho-phosphoric acid) with a flow rate of 1.0 ml/minute. The detection wavelength was set at 261 nm.

ANALYTICAL METHODOLOGY

Preparation of Reagents and Standards

Mobile phase

Precisely weighed and dissolved 2.6gms of potassium hydrogen phosphate and 0.6gms of dipotasssium hydrogen phosphate in 1000ml water , P^H was adjusted to 6.0 ±0.1 with dilute orthophosphoric acid solution.The above prepared buffer and methanol were mixed in the proportion of 40:60 v/v. The mobile phase was then duly filtered through 0.45 µm nylon membrane vacuum filtration and duly degassed by sonication.

Preparation of Desloratadine and Montelukast sodium stock and standard solutions

5mg of Desloratadine and 10 mg of Montelukast sodium were weighed accurately and transferred in to 50 ml volumetric flasks separately. 30 ml of 30ml of diluents were added added and sonicated to dissolve the compounds. These were made up to marks with diluent which yields concentrations of Desloratadine and Montelukast sodium 100µg/ml & 200µg/ml respectively (stock solution A). 10 ml of above solutions were pipetted out into 100ml volumetric flasks and volumes were made up to mark with diluent which gave concentrations of Desloratadine and Montelukast sodium 10µg/ml & 20µg/ml respectively (stock solution B). The standard solution ranging from 2.5-15 mL were transferred into a series of 100 ml volumetric flasks to provide a final concentration range of Desloratadine 2.5-15 µg/ml and Montelukast sodium 5-30 µg/ml, and the contents of each flask was made up to the mark with diluent.

Preparation of Formulation Test Solutions

Twenty tablets containing Desloratadine and twenty tablets containing Montelukast sodium were weighed and finely powered. An accurately weighed portion of the powder equivalent to 5 mg of Desloratadine and 10 mg Montelukast sodium was transferred into 50 ml volumetric flask. 30ml of diluents were added added and shaken for 20 minutes by manually and further sonicated for 10 minutes. This was diluted up to the mark with diluent. This solution was centrifuged at 8000 rpm for 10 minutes. The solution was filtered through 0.45 µm Nylon membrane filter paper .The supernatant solution was decanted into another test tube which yields concentrations of Desloratadine and Montelukast sodium 100µg/ml & 200µg/ml respectively .10 ml of supernatant solution was transferred into another 100 ml volumetric flask and made up to the mark with diluent which gave concentrations of Desloratadine and Montelukast sodium 10µg/ml & 20µg/ml respectively. 2.5-15 ml of solutions were transferred into a series of 100 ml volumetric flasks separately and made up to the mark with diluent. 20 µL of blank solution, placebo solution, three times of standard solutions were injected, disregarding peaks due to blank and placebo.

Assay procedure

The column was equilibrated for at least 30 minutes with mobile phase flowing through the system with a flow rate of 1.0 mL/min. Detector was set at a wavelength of 261nm. Twelve sets of the drug solutions were prepared in diluent containing Desloratadine and Montelukast sodium at a concentration range of 2.5 – 15 µg/mL and 5-30 µg/mL. Then 20 µl of each standard and sample solution were injected for Six times separately. The retention time for Desloratadine and Montelukast sodium succinate were found to be 2.460 and 3.129 minutes. It is shown in figure-3. The peak areas of the drug concentrations were calculated. The results are given in table-9.

System suitability solution

Desloratadine and Montelukast sodium standard working solution was used as system suitability solution.

Procedure

Equal volumes of blank were injected and twelve replicate injections of system suitability solutions in to column (Desloratadine and Montelukast sodium standard working solution).

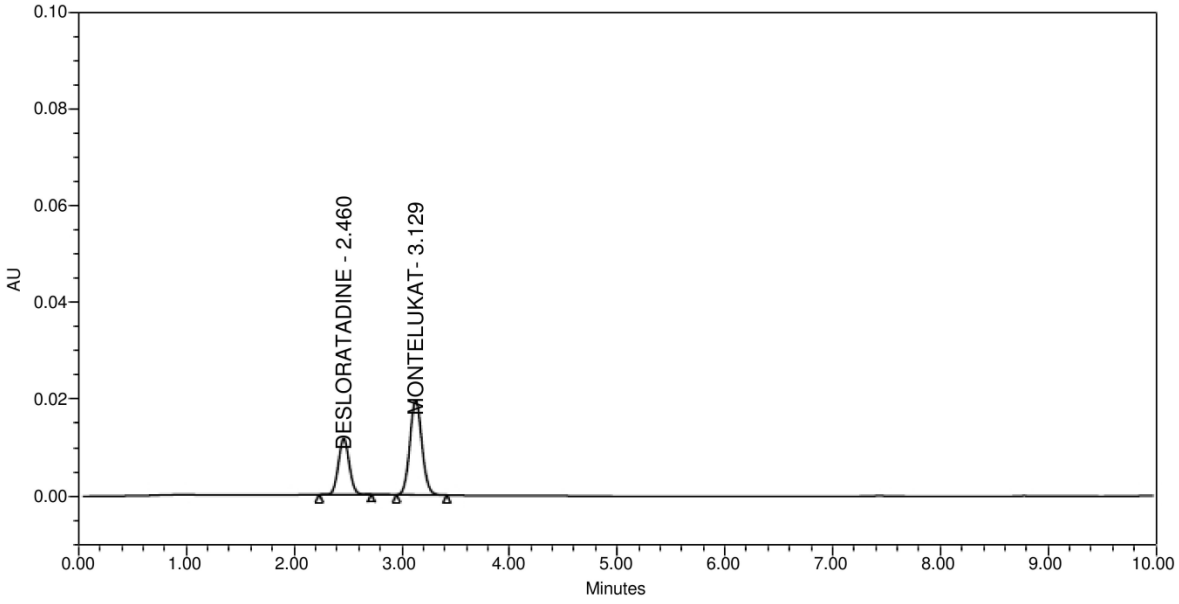
The chromatograms were recorded. Disregarded any peaks due to blank in the test solution. % RSD of twelve replicate injections of system was calculated (Desloratadine and Montelukast sodium standard working solution). Tailing factor and theoretical plates of the peak in the chromatogram obtained with 12th injection of system suitability solution (Desloratadine and Montelukast sodium standard working solution) were checked.

System suitability requirements from SST solution:

a) Tailing factor : NMT 2.0
b) Theoretical Plates : NLT 2000
c) Resolution : NLT 2.0

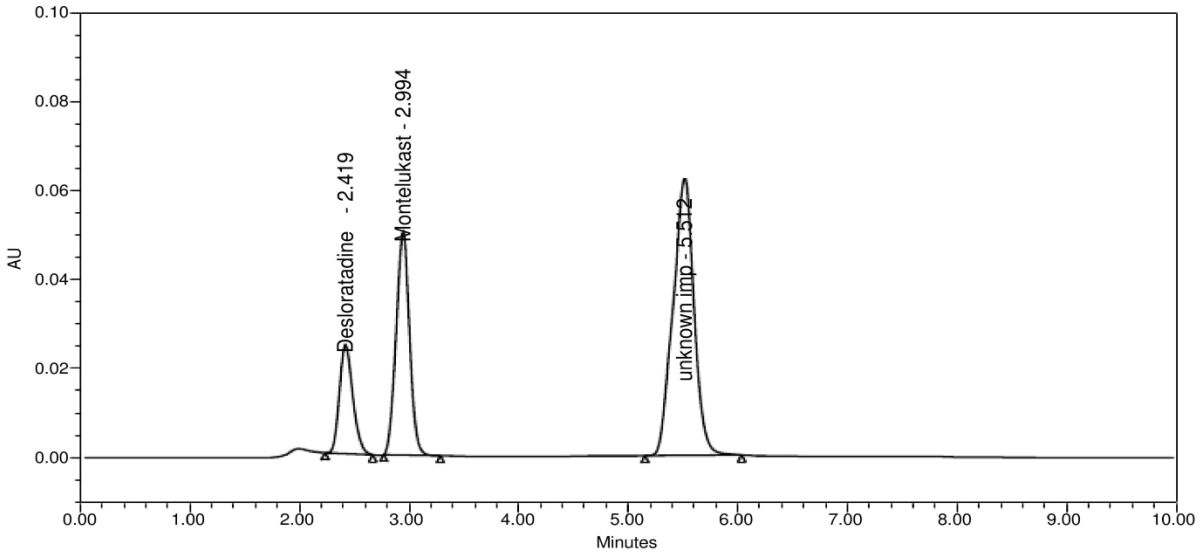
Linearity and Construction of Calibration Curve

Linearity of the peak area response was determined by taking measurements at twelve concentrations of working standard of Desloratadine and Montelukast sodium solutions in the range of 2.5-15 µg/mL and 5-30 µg/mL. 20µL quantity of the solution was injected each time in to the column. The drug elutes were monitored at 261 nm at a column temperature of 30ºC and the corresponding chromatograms were recorded. The Linearity of the calibration curve was plotted between the mean peak areas versus respective Concentration in figures-8 & 9.



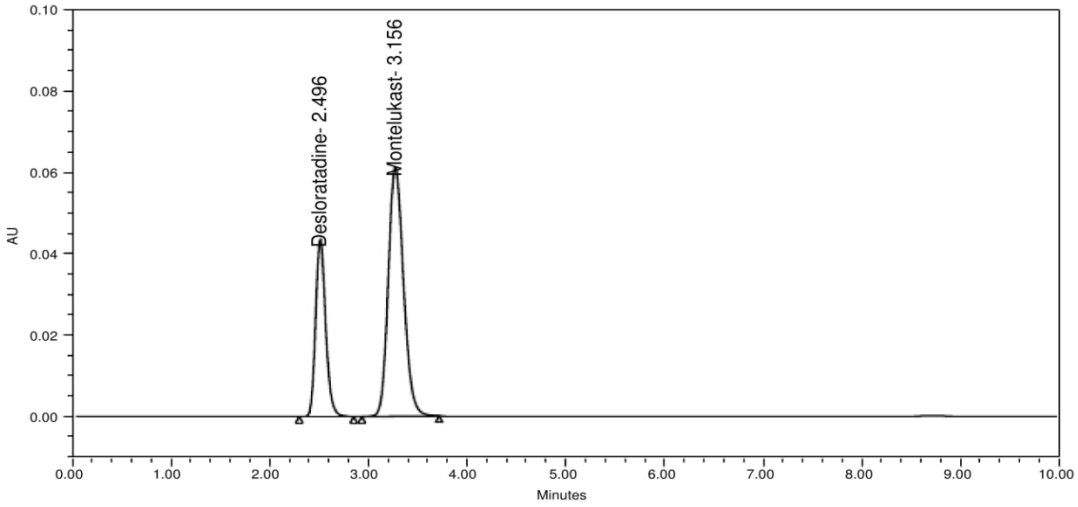
	Peak Name	RT	Area	% Area	Resolution	USP Tailing	USP Plate count
1	DESLORATADINE	2.460	80429	34.32		1.03	5635
2	MONTELUKAST	3.129	153932	65.68	2.42	1.08	4856

Fig.3: Chromatograms of Desloratadine and Montelukast sodium



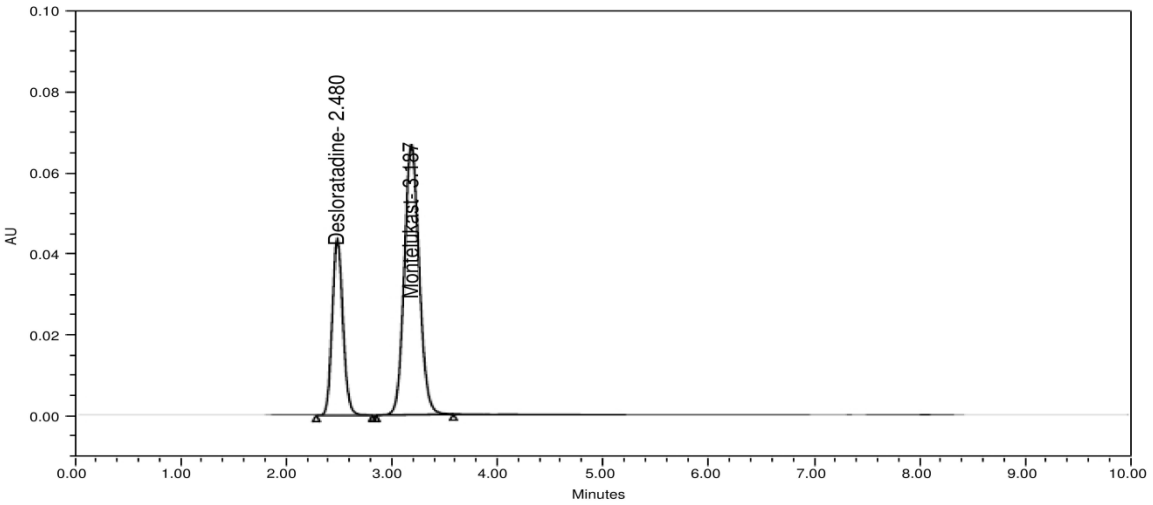
	Peak Name	RT	Area	% Area	Resolution	USP Plate Count	USP Tailing
1	Desloratadine	2.419	206595	14.13		5868.34	1.03
2	Montelukast	2.994	426501	29.18	2.38	4722.69	1.09
3	unknow n imp	5.512	828616	56.69	8.99	3391.32	0.97

Fig. 4: Chromatograms of Desloratadine and Montelukast sodium in acid degradation



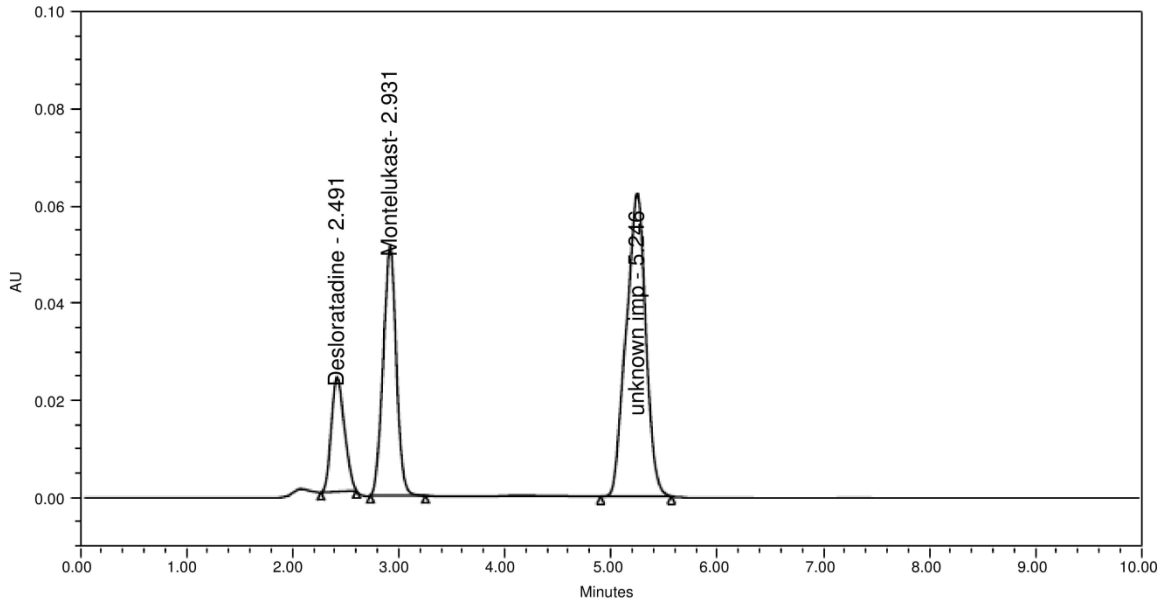
	Peak Name	RT	Area	% Area	Resolution	USP Tailing	USP Plate count
1	Desloratadine	2.496	301201	31.90		1.04	5656
2	Montelukast	3.156	634512	68.10	2.33	1.06	4889

Fig. 5: Chromatograms of Desloratadine and Montelukast sodium in base degradation



	Peak Name	RT	Area	% Area	Resolution	USP Tailing	USP Plate count
1	Desloratadine	2.480	302345	32.42		1.02	5859
2	Montelukast	3.187	642513	67.58	2.26	1.09	4812

Fig. 6: Chromatograms of Desloratadine and Montelukast Sodium in UVdegradation



	Peak Name	RT	Area	% Area	Resolution	USP Plate Count	USP Tailing
1	Desloratadin	2.491	200983	13.94		5710.84	1.02
2	Montelukast	2.931	445642	30.58	2.20	4550.57	1.10
3	unknown imp	5.246	829963	55.48	8.22	3133.58	0.95

Fig. 7: Chromatograms of Desloratadine and Montelukast sodium in Peroxide degradation

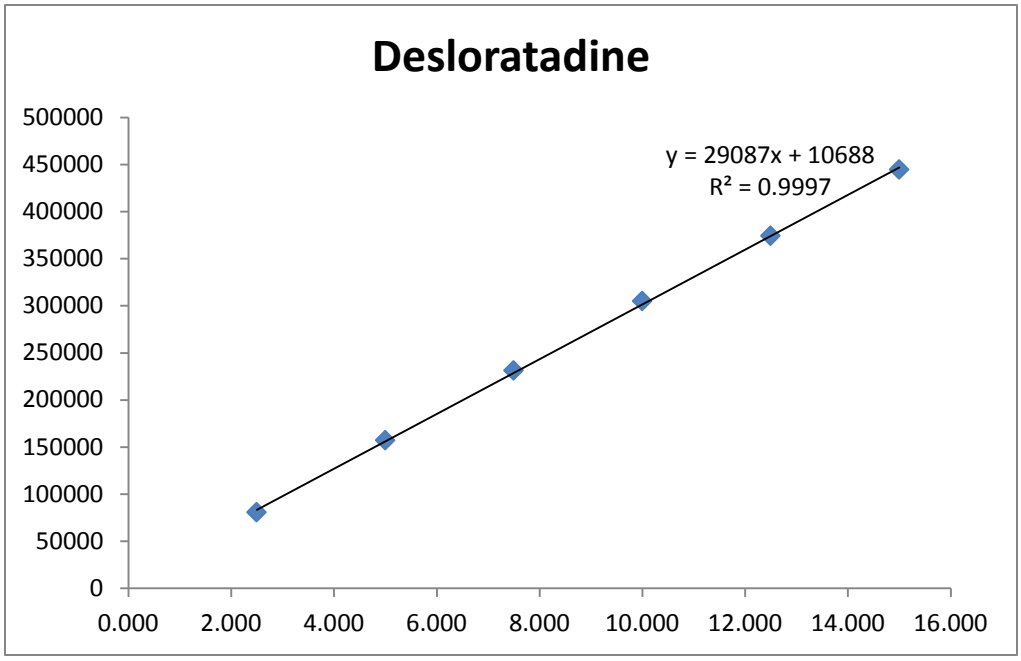


Fig. 8: Linearity Chromatogram of Desloratadine

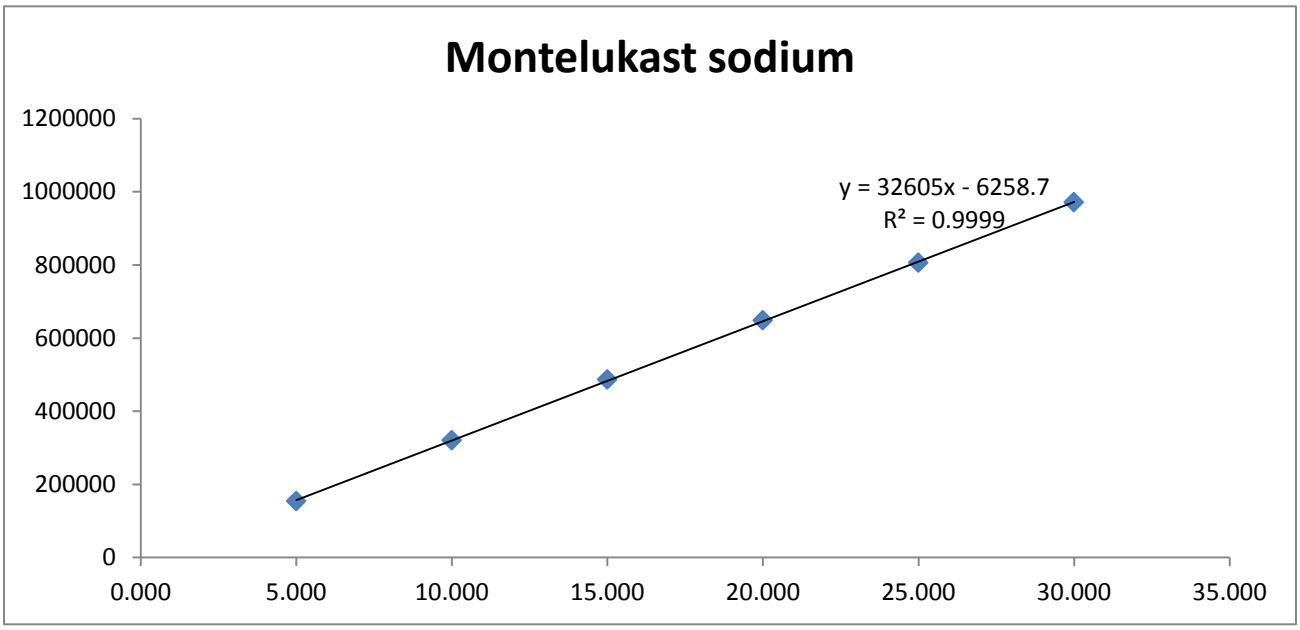


Fig. 9: Linearity Chromatogram of Montelukast sodium

Table -1: Performance calculations, detection characteristics precision and accuracy of the proposed method for Desloratadine and Montelukast sodium

Parameter	HPLC method for Desloratadine	HPLC method for Montelukast sodium
Wavelength (nm)	261	261
Retention times (t) min	2.460	3.129
Linearity range (µg ml^{-1})	2.5 – 15	5 - 30
LOD (µg ml^{-1})	0.081	0.084
LOQ (µg ml^{-1})	0.246	0.254
Regression equation	(y=bc+a)	(y=bc+a)
Slope (b)	29087	32605
Intercept (a)	10688	6258.7
Correlation coefficient(r^2)	0.9997	0.9999
Relative Standard deviation (%RSD)	0.1560	0.638
Intermediate Precision (%RSD)	0.30	0.57

%RSD of six independent determinations

Table-2: Results of linearity

Desloratadine		Montelukast sodium	
Conc. (µg)	**Area**	**Conc.(µg)**	**Area**
2.5	80429	5	153932
5	156831	10	320005
7.5	230829	15	486741
10	304779	20	648082
12.5	373895	25	806001
15	444417	30	971256

Table -3: System precision and system suitability

S No.	Desloratadine		Montelukast sodium	
	RT	Area	RT	Area
1	2.512	309068	3.272	659938
2	2.509	309542	3.268	661305
3	2.507	310421	3.265	661042
4	2.51	311140	3.269	659916
5	2.513	310041	3.271	659063
6	2.513	309983	3.273	659989
Avg	2.511	310032.5	3.27	660208.233
Std Dev	0.002	714.263	0.003	826.573
%RSD	0.096	0.23	0.09	0.125

Table -4: Method precision

S No	Desloratadine		Montelukast sodium	
	RT	Area	RT	Area
1	2.483	308049	3.185	651416
2	2.512	308780	3.272	659714
3	2.483	307916	3.185	651775
4	2.483	308041	3.185	651321
5	2.483	308049	3.185	652056
6	2.512	309067	3.272	659714
Avg	2.492	308317	3.214	654333
Std Dev	0.0149	481.1432	0.0449	4176.588
%RSD	0.5979	0.156	1.397	0.638

Table-5: Ruggedness of Desloratadine Day 1 and Day 2

S No	Name	RT	Area
1	Injection-1	2.491	308038
2	Injection-2	2.493	307849
3	Injection-3	2.489	309025
4	Injection-4	2.492	308452
5	Injection-5	2.505	310215
6	Injection-6	2.508	310566
7	Injection-7	2.483	308049
8	Injection-8	2.512	308780
9	Injectoion-9	2.483	307916
10	Injection-10	2.483	308041
11	Injection-11	2.483	308049
12	Injection-12	2.507	309067
	AVG	2.4945	308670
	STDEV	0.011619	911.2429
	%RSD	0.47	0.30

Table-6: Ruggedness of Montelukast sodium Day 1 and Day 2

S. No	Name	RT	Area
1	Injection-1	3.19	651775
2	Injection-2	3.193	650567
3	Injection-3	3.19	651352
4	Injection-4	3.196	651224
5	Injection-5	3.268	656563
6	Injection-6	3.271	659059
7	Injection-7	3.185	651466
8	Injection-8	3.272	659714
9	Injectoion-9	3.185	651775

10	Injection-10	3.185	651321
11	Injection-11	3.185	652056
12	Injection-12	3.272	659714
	AVG	3.216	653878
	STDEV	0.040593	3708.975
	%RSD	1.26	0.57

Table-7: Robustness study of Desloratadine and Montelukast sodium

S.No.	Peak Name	RT	Area	% Area	USP Plate Count	USP Resolution	USP Tailing
1	DESF-263nm	2.490	304658	32.36	5869	----	1.04
	MKT-263nm	3.190	649985	67.64	4811	2.33	1.1
2	DES -259nm	2.486	303345	32.23	5889	----	1.04
	MKT-259nm	3.191	656458	67.77	4802	2.30	1.1
3	DES Tem-25˚C	2.483	305546	32.26	5822	---	1.04
	MKTTem-25 ˚C	3.179	677490	67.74	4768	2.32	1.09
4	DES Tem-35˚C	2.481	301685	32.22	5896	---	1.04
	MKT Tem-35˚C	3.179	684125	67.88	4756	2.34	1.09

Table-8: Degradation study of Desloratadine and Montelukast sodium

Stress conditions	Time	Area		Assay of active		Deg%		Peak purity	
		DES	MKT	DES	MKT	DES	MKT	DES	MKT
Acid	24 hrs	206595	426501	66.82	64.51	33.18	35.49	1.0	1.0
Base	24 hrs	301201	634512	97.41	96.27	2.59	3.73	1.0	1.0
Peroxide	24 hrs	200983	445642	65	67.61	35	32.39	1.0	1.0
UV	10 days	302345	642513	97.78	97.48	2.22	2.52	1.0	1.0

Table-9: Assay Results of Desloratadine and Montelukast sodium

Drug	Amount present/tablet(mg)	Amount Found /tablet(mg)	% of Assay
Desloratadine	5	4.99	99.81
Montelukast sodium	10	9.937	99.37

Table-10: Accuracy data (Triplicate values at 50,100 &150 percent levels) of Desloratadine

S.No	Spike level	Peak area	Amount Added (µg/ml)	Amount Recovered (µg/ml)	%Recovery	Avg	% RSD
1	50%	156052	5	5.023	100.45	99.897	0.588
		155954	5	4.998	99.96		
		155056	5	4.964	99.28		
		308990	10	9.982	99.82	99.587	0.355

2	100%	308130	10	9.976	99.76		
		307986	10	9.918	99.18		
		460586	15	14.83	98.86	99.487	0.619
3	150%	462105	15	14.926	99.51		
		466650	15	15.013	100.09		

Table-11: Accuracy data (Triplicate values at 50,100 &150 percent levels) of Montelukast sodium

S.No	Spike level	Peak area	Amount Added (µg/ml)	Amount Recovered (µg/ml)	%Recovery	Avg	% RSD
		331025	10	9.996	99.96		
1	50%	330896	10	9.95	99.50	99.484	0.488
		329563	10	9.899	98.99		
		658865	20	19.972	99.86		
2	100%	658344	20	19.998	99.99	99.75	0.311
		657949	20	19.88	99.40		
		981302	30	29.643	98.81		
3	150%	985455	30	29.865	99.55	99.297	0.425
		989089	30	29.859	99.53		

METHOD VALIDATION

Specificity study

Mobile phase along with placebo were injected to check the interference at the retention time of Desloratadine and Montelukast sodium in the established chromatographic condition and no interference were observed at the designated Retention Time which was established by peak purity of the chromatogram by UV-Vis detector.

Stress studies

Acid, Alkaline, Peroxide and UV degradation studies were conducted and Desloratadine and Montelukast sodium were subjected to this condition. 1N HCl, 0.1N NaOH, 3% hydrogen peroxide and UV for 10 days were used for stress testing studies. The samples are neutralized before injecting into the system for acid and alkaline samples. Oxidative and UV samples were injected after proper dilution as such. Placebo and mobile phase were also subjected to same treatment as sample to check for interferences.

Precision

ICH describes precision as closeness of individual measure of analytes when the procedure is applied repeatedly to multiple times interday and intraday precision has been established in the method.

Accuracy

It was evaluated at three levels of 50%, 100% and 150% of test concentration by adding known amount of drug to placebo and extracting the sample. Three sets were prepared and analyzed.

Solution stability

Desloratadine and Montelukast sodium and their formulation stabilities were carried out for a period of 48 hours at auto sampler at 25^0c temperature.

Robustness

Varying conditions of wavelength, column temperature were carried out as per ICH guidelines to estimate the effects on the method.

RESULTS AND DISCUSSIONS

Method development and optimization

Actual chromatographic conditions were established after number of preliminary experiments for selecting the proper mobile phase system. Different mobile phase systems were tested, and selection of the proper system depended on its ability to give good separation between the pure drugs and their possible degradation products. Acceptable separation was achieved on Zodiac C18,

100x4.6, 5µ using a mobile phase composed of mixed buffer and Methanol in the ratio 40: 60 (pH was adjusted to 6.0 ±0.1 by using Ortho-phosphoric acid) pumped with a flow rate of 1.0 ml/min the column temperature was kept constant at 30ºC. Under these chromatographic conditions, the run time sample was 10 min, and the retention times of Desloratadine and Montelukast sodium 2.460 and 3.129 min. The representative chromatogram is shown in figure-3.

Performance calculations, detection characteristics precision and accuracy of the proposed method for Desloratadine and Montelukast sodium were reported in the table-1.

System suitability

System suitability parameters like theoretical plates per meter, tailing factor, percentage relative standard deviation of area and retention time of twelve injections were carried out and the values are well within the limits as shown in Table -3.

Linearity and sensitivity

A linear calibration plot Desloratadine and Montelukast sodium was constructed at nine point concentration levels 2.5-15 µg/ml and 5-30 µg/ml in duplicate. Average peak area of Desloratadine and Montelukast sodium were plotted against respective concentrations and linear regression analysis was performed. Correlation coefficient was found to be r^2=0.9997 and r^2=0.9999 respectively. Limit of detection (LOD) and limit of quantification (LOQ) values for Desloratadine was 0.081µg/ml and 0.246µg/ml and for Montelukast sodium was 0.084 µg/ml and 0.254µg/ml respectively. Results are given in table- 2.

Precision

The precision of the assay method was evaluated for repeatability and intermediate precision. For intra-day precision and inter-day precision, the percentage relative standard deviation of Desloratadine and Montelukast sodium was found to be 0.1560% and 0.638% respectively. These values were well within the acceptable limit of 2%, as per USP. Results are given in table- 4.

Accuracy

Known amount of standard was spiked in 50%, 100%, 150% concentration in triplicate to test solution and recovery of drug was calculated. The accuracy of method was established at three concentration levels at 5, 10 and 15 µg/ml of Desloratadine and 10, 20 and 30µg/ml of Montelukast Sodium standards. The recoveries at three different concentrations were found to be within the range of 95.0 to 105 % as per ICH guidelines. Mean % recovery was found between 99.297 to 99.897. The results are given in tables-10 & 11.

Robustness

The robustness of assay method was studied by incorporating small but deliberate changes in the analytical method (variations in wave length and column temperature) and also by observing the stability of the drugs for 48 hours at room temperature in the dilution solvent. In all the varied chromatographic conditions, there was no significant change in chromatographic parameters. Results are given in table- 7.

STRESS STUDIES

Stress testing of the drug substance can help in identify the likely degradation products, which can in turn help to establish the degradation pathways and the intrinsic stability of the molecule

Acid degradation studies

5mg of the DES and 10mg of MKT were weighed accurately and transferred to 50 ml volumetric flask, added 30 ml of freshly prepared 1N HCl and kept at room temperature for 24 hours. After keeping the solution for 24 hrs at room temperature, filtered and then neutralized the solution up to the volume with 0.1 N NaOH. Diluted 10 ml of the above solution to 100 ml with diluent. 20µl of the solution was injected into the chromatographic system and the chromatogram was recorded as shown in figure- 4. The results are given in table-8.

Base degradation studies

5mg of the DES and 10mg of MKT were weighed accurately and transferred to 50 ml volumetric flask, added 30 ml of freshly prepared 0.1N NaOH and kept at room temperature for 24 hours. After keeping the solution for 24 hrs at room temperature filtered and then neutralized the solution up to the volume with 1 N HCl. Diluted 10 ml of the above solution to 100 ml with diluent. 20µl of the solution was injected into the chromatographic system and the chromatogram was

recorded as shown in figure -5. The results are given in table-8.

Peroxide degradation

5mg of the Desloratadine and 10mg of Montelukast sodium were weighed accurately and transferred to 50ml volumetric flasks containing 30 ml 3% H_2O_2 and kept at room temperature for 24 hours. After keeping the solution for 24 hrs at room temperature, it was filtered. 10 ml of the above solution was diluted to 100 ml with diluent. 20µl of the solution was injected into the chromatographic system and the chromatogram was recorded as shown in figure -7. The results are given in table-8.

UVdegradation

Sufficient amount of Desloratadine and of Montelukast sodium powder was transferred into petridish spread evenly for NMT 1mm thickness and kept inside hot air oven at 40˚C for 10 days. Samples were collected at different time intervals and final dilution were done with the mobile phase and loaded into HPLC system. The representative chromatogram is shown in fig-6. The results are given in table-8.

CONCLUSION

A simple and precise stability indicating RP-HPLC method has been developed for determination of Desloratadine and Montelukast sodium in tablet dosage form. The %RSD values in precision, recovery studies, robustness and ruggedness studies was found less than 2.0% which indicates that the method is precise, accurate and robust. Limit of detection (LOD) and limit of quantification (LOQ) values for Desloratadine was 0.081 and 0.246µg/ml and for Montelukast sodium was 0.081 and 0.254 µg/ml respectively. This indicates that the method is sensitive for the determination of lower concentration of the both the drugs. The % assay was found to be well within the acceptable limit. The percentage recoveries of MKT and DES were in the range of 99.40%-99.99%and 99.18%-100.45% respectively .DES and MKT were highly susceptible to acid (33.18 & 35.49), peroxide conditions (35 & 32.39) and least susceptible to UV (2.22 & 2.52) and basic conditions (2.59 & 3.73).The peaks of the degradants in each condition were well separated from main peaks. Purity plot confirmed that there is no interference of any degradants at the retention time of the main peaks indicates that the developed method is stability indicating. The proposed method can be used as an alternative method for the analysis of Desloratadine and Montelukast sodium in its dosage forms.

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