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Method Development and Validation for Simultaneous Estimation of Dexlansoprazole and Meloxicam by Rp-Hplc

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Abstract

To develop a method for simultaneous estimation of Dexlansoprazole and Meloxicam by RP-HPLC and validate the developed method according to ICH guidelines. The column used for method development of Dexlansoprazole and Meloxicam was Hypersil-BDS, C18, 250*4.6 mm, 5µ. Methanol and Acetonitrile were used in the ration of 60:40 for the method development. The developed method was validated for various parameters such as specificity, linearity, range, accuracy, precision, system suitability, robustness, ruggedness etc. The method was developed and optimized method was chosen and validated and the results were tabulated according to ICH guidelines. The result obtained in this study demonstrated that the HPLC method described is specific, accurate, precise, linear, ruggedness, robustness and stability indicating for the simultaneous determination of Dexlansoprazole and Meloxicam.

Keywords: Meloxicam; Dexlansoprazole; HPLC

Introduction

Method validation can be defined as (ICH) Establishing documented evidence, which provides a high degree of assurance that a specific activity will consistently produce a desired result or product meeting its predetermined specifications and quality characteristics [1-7]. Method validation study include system suitability, linearity, precision, accuracy, specificity, robustness, limit of detection, limit of quantification and stability of samples, reagents, instruments.

System suitability

Before starting any experiment, the operator should daily check the functioning of the HPLC so as to be sure that the system is capable of providing results of high accuracy [8].

Linearity

Linearity is defined as the measure of the proximity of the straight line obtained from a calibration plot of response vs. concentration. It can be obtained by performing the experiments using different concentrations of the analyte [9].

Precision

Precision is the degree of similarity between individual results when the same test procedure is applied to different samplings of a same sample [10].

Accuracy

The accuracy is the closeness of an obtained value to the true value. A method with high accuracy, the measured value is identical to the true value [11].

Specificity/Selectivity

According to ICH, the term specific indicates a method producing results for a single analyte while the term selective indicates a method producing results for a number of analytes [12].

Robustness

According to ICH robustness is a measure of the capacity of a method to remain unaffected by small, variations in parameters used in the method.

Limit of detection

LOD is the least concentration of an analyte in a sample that can be detected, but not quantified using specific experimental conditions [13].

Limit of quantification

LOQ is the least concentration of an analyte that can be detected with preferable precision and accuracy using specific experimental conditions.

Materials and Methods

Instruments

HPLC-Waters 2690/5 with PDA Detector, Software-Empower

Electronic balance-Mettler Toledo Model XS-205DU

Sonicator (FAST CLEAN)

Column details

Hypersil-BDS, C18, 250*4.6 mm, 5 µ

Reagents

Purified water (Milli-Q)

Methanol HPLC Grade

Acetonitrile

Raw material

Dexlansoprazole and Meloxicam working Standards

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Method Development for HPLC

Trail 1

Mobile Phase: Degassed Methanol: Water in the ratio of 90:10V/V

Observation: No proper shape of the peaks and extra peaks observed.

Trail 2

Mobile Phase: Degassed Methanol: Water in the ratio of 80:20V/V

Observation: Asymmetric peaks were observed.

Trail 3

Mobile Phase: Degassed Methanol: Water in the ratio of 40:60V/V

Observation: Asymmetric peaks were observed and efficiency of separation is not good.

Trail 4

Mobile Phase: Degassed Methanol: Water in the ratio of 50:50V/V

Observation: Peak fronting is seen in both the peaks.

Trail 5

Mobile Phase: Acetonitrile: Water in the ratio of 50:50V/V

Observation: Peak splitting is seen and peak tailing is seen.

Trail 6

Mobile Phase: Methanol: Acetonitrile in the ratio of 90:10V/V

Observation: Sharp peaks are not observed.

Trail 7

Mobile Phase: Methanol: Acetonitrile in the ratio of 80:20V/V

Observation: Peak tailing is seen and no sharp peaks are observed.

Trail 8:

Mobile Phase: Methanol: Acetonitrile in the ratio of 70:30V/V

Observation: Good resolution is seen but sharp peaks are not obtained.

Trail 9: Optimized Method

Mobile Phase:

Observation: Good resolution and sharp peaks with good elution are observed (Figure 1) (Table 1).

Method Validation

Analytical Method Validation Parameters

This Validation describes the procedure by HPLC as per ICH Guidelines (Q2B). The method validation parameters include:

Specificity

Linearity

Accuracy

Range

Precision

Intermediate precision (ruggedness)

Method precision

Robustness

System suitability

Results and Discussion

The chromatographic method development for the estimation of Meloxicam and Dexlansoprazole were optimized by several trials for various parameters as different column, flow rate and mobile phase, finally the following chromatographic method was selected for the quantification of Meloxicam and Dexlansoprazole by RP-HPLC [14-16].

Validation Report

Specificity

The method shows excellent specificity with Meloxicam and Dexlansoprazole eluting at retention of 2.95 minutes and 4.19 minutes respectively. No interference was observed with mobile phase [17-19].

Linearity

The linearity study was performed and the correlation coefficient of Meloxicam and Dexlansoprazole were found to be 0.9997 and 0.9995 respectively (NMT 0.999).

Accuracy

The accuracy study was performed for % recovery. The % recovery was found to be 99.7% to 100.6% for Meloxicam. (NLT 98% and NMT 102%).

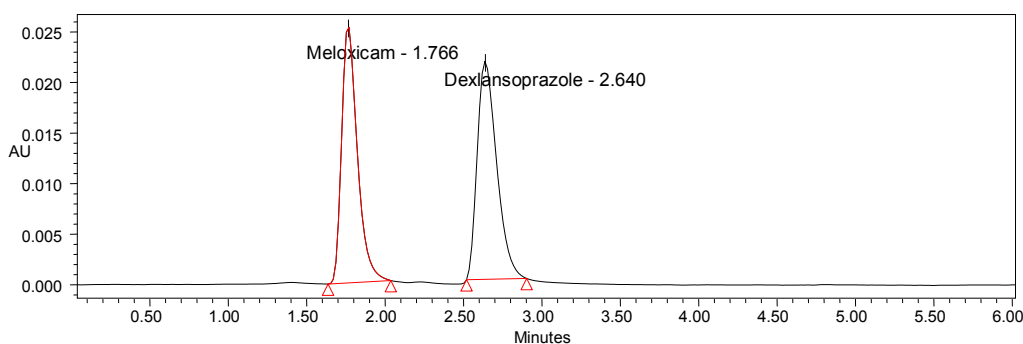The accuracy study was performed for % recovery. The % recovery

Figure 1: Optimized method-Methanol: Acetonitrile in the ratio of 60:40V/V.

	Name	Retention Time	Area	% Area	Height	USP Resolution	USP Tailing	USP Plate Count
1	Meloxicam	1.766	177126	48.75	25253		1.389748	1522.681250
2	Dexlansoprazole	2.640	186204	51.25	21571	4.116883	1.403226	2052.683951

Table 1: Trail 9 Methanol: Acetonitrile in the ratio of 70:30V/V.

was found to be 99.9 % to 100.4% for Dexlansoprazole. (NLT 98% and NMT 102%).

Summary and Conclusion

The analytical method was developed by studying different parameters. Isobistic point of wavelength for both the drugs was set at 248nm and the peaks purity was excellent. Injection volume was selected to be 20 μl which gave a good peak area. The column used for study was Hypersil C18 BDS, chosen good peak shape. Ambient temperature was found to be suitable for the nature of drug solution. The flow rate was fixed at 1.0 ml/min because of good peak area, satisfactory retention time and good resolution. Different ratios of mobile phase with ratio of 60:40 methanol: acetonitrile was fixed due to good symmetrical peaks and for good resolution. So this mobile phase was used for the proposed study.The result obtained in this study demonstrated that the HPLC method described is specific, accurate, precise, linear, rugged, robust and stability indicating for the determination of assay of Meloxicam and Dexlansoprazole. Therefore, the method is suitable for intended uses.

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