

HPLC method development and validation for the simultaneous estimation of Dexlansoprazole and Domperidone in capsule dosage form

Nithyasri*, Dr. Vaijayantimala P, Dr. S Vijaya Bhaskar, Dr. Shachindra L Nargund, Harshitha K B, Nithyashree H P

Department of Pharmaceutical Quality Assurance, Nargund college of Pharmacy, Bengaluru, Karnataka, India

Corresponding Author: Nithyasri

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Abstract

A simple, rapid, accurate, precise, and economical RP-HPLC method was developed and validated for the estimation of Dexlansoprazole (DXL) and Domperidone (DOM) in pharmaceutical dosage forms. Chromatographic separation was achieved using a Shim-pack C8 column with HPLC grade Acetonitrile: Water (pH adjusted to 3.0, 1% Ortho Phosphoric acid) (40:60 % v/v) as the mobile phase at a flow rate of 1.0 mL/min with UV detection at 210 nm. The method showed linearity over the concentration 20–100 µg/mL for Dexlansoprazole and 10–50 µg/mL for Domperidone with R² value of 0.9983 and 0.9932. Accuracy was confirmed by recovery studies, with recoveries of 98.53–101.25% for DXL and 99.85–101.76% for DOM. Precision showed %RSD below 2%. The method was also validated for LOD, and LOQ according to ICH guidelines and was suitable for routine analysis.

Keywords: Dexlansoprazole, Domperidone, Simultaneous Estimation, RP-HPLC, GERD, ICH Guidelines

Introduction

Disease: Gastro Esophageal Reflux Disease (GERD) is a prevalent condition affecting the upper gastrointestinal tract, characterized by the backward flow of stomach contents into the esophagus. Common symptoms include heartburn, bloating, nausea, and vomiting. The increased vulnerability to anxiety and depression among GERD patients stems from factors like acid reflux, heartburn, inherited susceptibility and quality of life [1].

Dexlansoprazole: Dexlansoprazole is a new-generation Proton Pump Inhibitor (PPI) used for the management of symptoms associated with GERD and erosive esophagitis. Dexlansoprazole is the R-enantiomer of Lansoprazole. Compared to the older generation of PPI, Dexlansoprazole has a unique pharmacokinetic profile due to its delayed-release and dual-delivery release system. Dexlansoprazole inhibits the final step in gastric acid production by blocking the (H⁺, K⁺) ATPase enzyme [2].

Domperidone: Domperidone acts as a gastrointestinal emptying (delayed) adjunct and peristaltic stimulant. The gastroprokinetic properties of Domperidone are related to its peripheral dopamine receptor blocking properties. Domperidone facilitates gastric emptying and decrease small bowel transit time by increasing esophageal and gastric peristalsis and by lowering esophageal sphincter pressure [3].

A fixed-dose combination of Dexlansoprazole and Domperidone is currently commercially available. Therefore, this combination was selected for the present study based on published clinical research demonstrating that the concurrent administration of both drugs produced improved therapeutic outcomes and yielded positive clinical results.

Analytical method validation:

In the absence of an established standard analytical method for the simultaneous estimation of Dexlansoprazole and Domperidone, a new analytical method was developed. The development of a suitable analytical method helps minimize analysis time and cost while ensuring accurate determination of both drugs. The developed method was optimized and subsequently validated through preliminary studies to confirm its accuracy, precision, reliability, and suitability for routine analysis [4].

The present study was undertaken to develop and validate a RP-HPLC method for the simultaneous determination of Dexlansoprazole (Fig. 1) and Domperidone (Fig. 2) in active pharmaceutical ingredients (APIs) and pharmaceutical dosage forms. The method was developed and validated in accordance with the ICH guidelines. Validation parameters including solubility, selection of mobile phase, selection of wavelength, linearity, range, accuracy, precision, limit of detection (LOD), and limit of quantification (LOQ), Repeatability were evaluated to establish the reliability and suitability of the proposed analytical method [5].

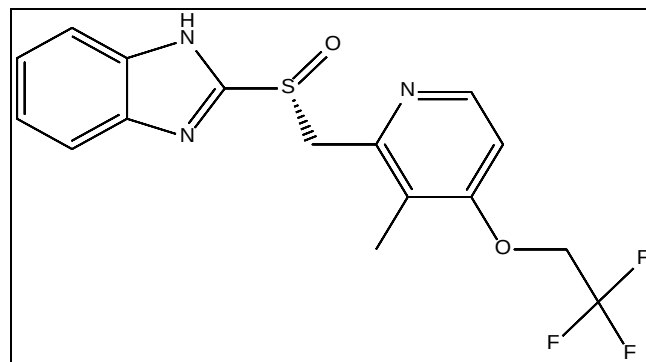


Fig 1: Dexlansoprazole [6]

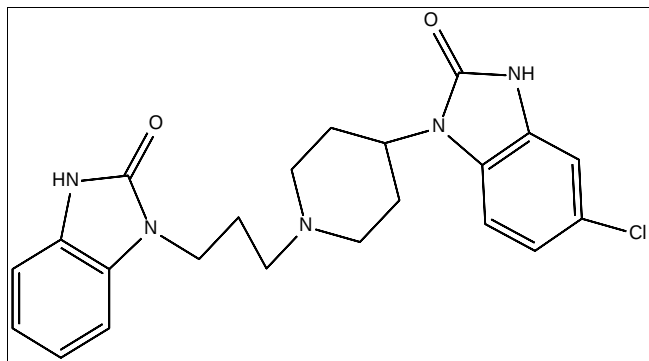


Fig 2: Domperidone ^[7]

Literature survey revealed that a number of methods have been reported for estimation of DEXLANSOPRAZOLE and DOMPERIDONE individually with other drugs. However, there is very few analytical methods including UV Spectroscopy ^[8-10], and no HPLC methods ^[11-13] reported for simultaneous analysis of these drugs in a combined dosage formulation.

Materials and Methodology

1. Materials

DXL and DOM API were procured as a gift sample from MSN Laboratories Private Ltd Hyderabad, Telangana, and Walter Healthcare Private Ltd Punjab, respectively. Marketed formulation DDR-D capsule is manufactured by MSN Laboratories Private Ltd, were procured from a local pharmacy in Bangalore, India, and used for the present study. HPLC-grade methanol and acetonitrile, procured from Avantor Performance Materials India Pvt. Ltd., Thane, Maharashtra, India, were used as solvents throughout the study. Double-distilled water was used for all analytical procedures.

Chromatographic analysis was performed using a Shimadzu Liquid Chromatograph (LC-20AT) equipped with a binary pump, UV-Visible detector (Shimadzu SPD-20A), Rheodyne injector (Model 7725i, 20 μ L loops), and LabSolutions software (Version 5.90). Separation was carried out using a Shimadzu Shim-pack C8 column (4.6 $\times$ 250 mm, 5 μ m). A Hamilton syringe (25 μ L) was used for sample injection. The mobile phase and sample solutions were filtered through Nylon membrane filters (25 mm, 0.2 μ m) and degassed using an ultrasonic cleaner (RC Systems MU1700). A Shimadzu UV-1700 UV-Visible spectrophotometer with UV Probe software (Version 2.32) was used for absorbance measurements, while a Value High Reliability Vacuum Pump was used for filtration. All glassware used was made of borosilicate glass and calibrated before use. Chemicals were weighed using a calibrated Sartorius TE-214S digital analytical balance (maximum capacity 200 g).

2. Methodology

2.1 Selection of Analytical Wavelength:

To determine the appropriate analytical wavelength for the simultaneous estimation of DXL and DOM, standard solutions of DXL (10 μ g/mL) and DOM (20 μ g/mL) were prepared using HPLC-grade water. The solutions were scanned individually over the UV range of 200–400 nm, and the overlaid spectra were evaluated to select the analytical wavelength. The overlay spectra are shown in Figure 3.

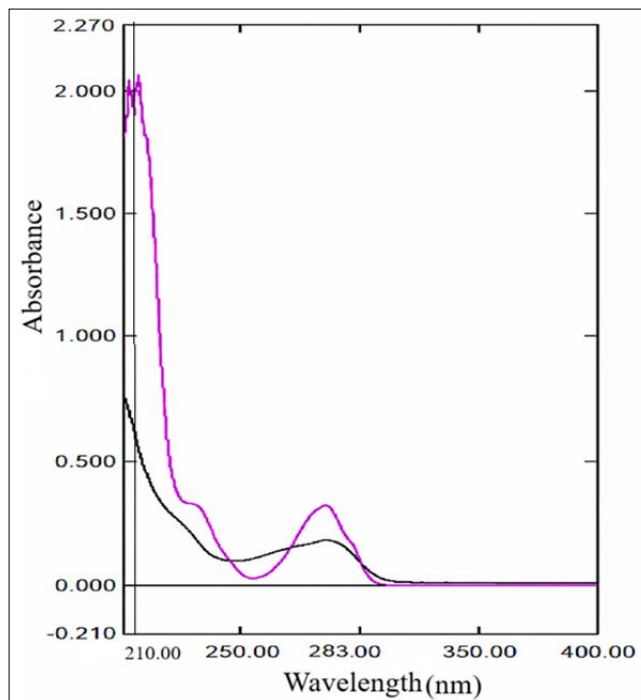


Fig 3: Overlay spectra of DXL (10 μ g/ml) and DOM (20 μ g/ml)

2.2 Finalized Chromatographic Conditions

- **Analytical Column:** Shim pack C8(250 mm $\times$ 4.6 mm, 5 μ m)
- **Mobile Phase:** HPLC grade ACN: Water (pH adjusted to 3.0, 1% Ortho Phosphoric acid) (40:60 % v/v)
- **Injection volume:** 20 μ l
- **Flow rate:** 1 ml/min
- **Detection Wavelength:** 210 nm
- **AUFS:** 0.1000
- **Pressure** $\sim$ 11.6 MPa

2.3 Preparation of Mobile Phase

The mobile phase was prepared by mixing 400 mL of HPLC-grade acetonitrile with 600 mL of Water (pH adjusted to 3.0). The prepared mobile phase was filtered through a 0.2 μ m membrane filter under vacuum and sonicated for 10 minutes before use.

2.4 Preparation of Standard Stock Solution

2.4.1 Preparation of Standard Solution of Dexlansoprazole:

An accurately weighed quantity of 10 mg of Dexlansoprazole was transferred into a 10 ml volumetric flask and dissolved in HPLC-grade methanol. The volume was made up to the mark with the same solvent to obtain a standard stock solution containing 1000 μ g/ml, designated as Std Stock DXL (A). Subsequently, 1 ml of this stock solution was transferred into a 10 ml volumetric flask and diluted to volume with HPLC-grade water to obtain a working standard solution of 100 μ g/ml, designated as Std Stock DXL (B).

2.4.2 Preparation of Standard Solution of Domperidone:

An accurately weighed quantity of 10 mg of Domperidone was transferred into a 10 ml volumetric flask and dissolved in HPLC-grade methanol. The volume was then made up to the mark with the same solvent to obtain a standard stock solution containing 1000 μ g/ml, designated as Std Stock

DOM (A). Thereafter, 1 ml of this stock solution was pipetted into a 10 ml volumetric flask and diluted to the mark with HPLC-grade water to obtain a working standard solution of 100 µg/ml, designated as Std Stock DOM (B).

2.5 Preparation of Calibration Curve for DXL and DOM

Aliquots of 2.0, 4.0, 6.0, 8.0 and 10 ml were pipetted from Std Stock DXL (B) into five separate 10 ml volumetric flasks. Similarly, 1.0, 2.0, 3.0, 4.0 and 5.0 ml aliquots were transferred from Std Stock DOM (B) into another set of volumetric flasks. The flasks were labelled as C1, C2, C3, C4, and C5 and the volume was made up to the mark with HPLC-grade water to obtain final concentrations of 20-100 µg/ml for DXL and 10-50 µg/ml for DOM.

The prepared solutions were filtered through a 25 mm, 0.25 µm nylon syringe filter and 20 µl of the solution was injected into the Rheodyne injector of the HPLC system. Chromatograms were recorded under the optimized chromatographic conditions after achieving a stable baseline. The peak areas of both drugs were measured for each concentration.

The overlay chromatograms of the calibration solutions are presented in Figure No. 4. Calibration curves for DXL and DOM were constructed by plotting peak area versus concentration, as shown in Figure No. 5 and Figure No. 6. The results of the calibration study are summarized in Table No. 1 and Table No. 2.

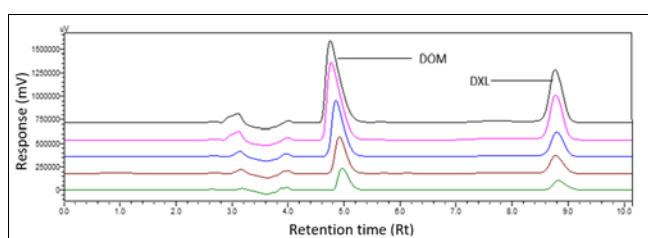


Fig 4: Overlay Chromatograms of DXL (20-100 µg/ml) and DOM (10-50 µg/ml) at 210 nm.

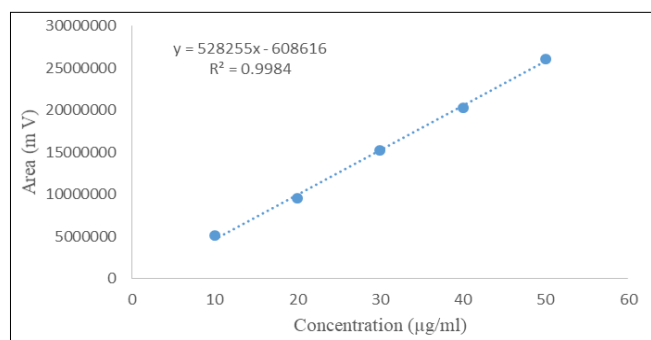


Fig 5: Calibration Curve of DXL at 210 nm.

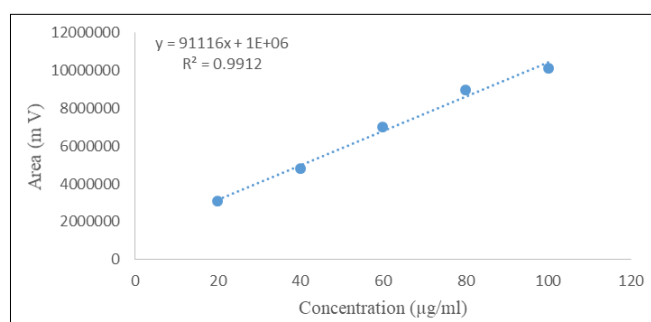


Fig 6: Calibration Curve of DOM at 210 nm.

2.6 Analysis of capsule formulation:

Twenty capsules of DXL and DOM (DDR-D, Dexlansoprazole 60mg and Domperidone 30mg manufactured by MSN Laboratories Pvt. Ltd) were weighed (filled Capsule) and capsule granules are crushed to obtain fine powder. An accurately weighed capsule powder equivalent to 10 mg of DOM and 20 mg of DXL was transferred into a 25 mL volumetric flask. 15 ml of HPLC-grade methanol was added and sonicated for 10 min. The volume was made up to the mark with the same solvent to get a concentration of DOM 400 µg/ml and DXL 800 µg/ml. The solution was filtered using Whatman filter paper No. 41, labelled as 'Sample Stock A'. Subsequently, 2.5 mL of Sample Stock A was transferred into a 10 mL volumetric flask and diluted to volume with methanol to obtain a working sample solution containing 100 and 200 µg/mL of Domperidone and Dexlansoprazole, which was labelled as Sample Stock B. From the 'Sample Stock B' 2 ml of aliquot is pipetted out and transferred to 10 ml of volumetric flask and make with HPLC grade water to obtain 20 µg/ml of DOM and 40 µg/ml of DXL.

The final sample solution was filtered through a 25 mm, 0.2 µm nylon syringe filter before analysis. A 20 µL aliquot of the solution was injected into the Rheodyne injector of the HPLC system using a Hamilton syringe. Chromatograms of sample solution was recorded under the optimized chromatographic conditions after obtaining a stable baseline. The peak areas of Dexlansoprazole and Domperidone were measured, and the drug content was determined using the single-point calibration method. The assay results are presented in Table No. 4, and the corresponding chromatograms are shown in Figure No. 7.

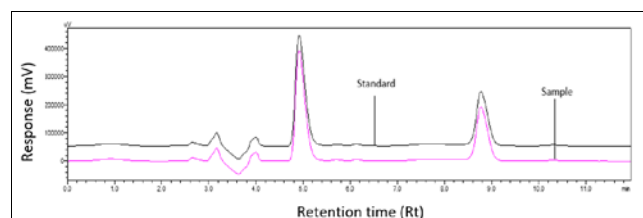


Fig 7: Overlay Chromatograms of Sample and Standard Solution of DXL and DOM at 210 nm.

3. Validation of RP-HPLC Method

3.1 Precision

The precision of the developed analytical method was evaluated by assessing repeatability.

3.2 Accuracy

The accuracy of the developed method was evaluated using the recovery study. Twenty tablets were accurately weighed, powdered, and used for analysis. Recovery studies were performed by spiking the pre-analysed sample with known concentrations of standard DXL (48, 60, and 72 µg/mL) and standard DOM (24, 30, and 36 µg/mL) at three concentration levels, namely 80%, 100%, and 120%. The percentage recovery was calculated for each level, and the results are presented in Table Nos. 10 and 11.

3.3 Linearity and Range

The linearity of the method was evaluated by preparing and analysing DXL standard solutions in the concentration range of 20-100 µg/mL and DOM standard solutions in the concentration range of 10-50 µg/mL. The linearity of the

method was established based on the correlation coefficient ($R^{[2]}$) obtained from the calibration curves.

3.4 Limit of Detection (LOD) and Limit of Quantitation (LOQ)

The limit of detection (LOD) and limit of quantitation (LOQ) were determined using the standard deviation of the y-intercepts of six calibration curves and the average slope of the corresponding calibration curves. The values were calculated using the following equations:

$$LOD = 3.3 \times \frac{\text{Standard Deviation of } y\text{-Intercepts of Six Calibration curves}}{\text{Average Slope of Six Calibration Curves}}$$

$$LOQ = 10 \times \frac{\text{Standard Deviation of } y\text{-Intercepts of Six Calibration curves}}{\text{Average Slope of Six Calibration Curves}}$$

The calculated LOD and LOQ values are presented in Table No. 3.

Results

Table 1: Results of Calibration Curve of DXL at 210 nm

Conc (µg/mL)	Peak area	SD	%RSD
10	3090066	3091175	3108286
20	4799157	4898266	4777268
30	7003357	7114568	7098654
40	8959088	8968177	8915935
50	10121703	10161211	10212541

Table 2: Results of Calibration Curve of DOM at 210 nm.

Conc (µg/mL)	Peak area	SD	%RSD
20	5044607	55118.23	1.081173
40	9508253	42158.44	0.442208
60	15228252	58773.91	0.384796
80	20305548	81661.83	0.400747
100	26079220	89829.52	0.343815

Table 3: Linear Regression Analysis of Calibration Curve for DXL and DOM at 210 nm.

Parameters	DXL	DOM
Beer's law limit (µg/ml)	20 to 100	10 to 50
Wavelength (nm)	210	210
(Correlation Coefficient) $R^{[2]}$	0.9906	0.9983
Slope	90832	94745
Intercept	529024	588642
LOD (µg/ml)	2.59	0.457
LOQ (µg/ml)	7.85	1.385

Table 4: Assay Results of Capsule Formulation.

Amount Present(µg/ml)		Amount Found(µg/ml)		%ASSAY	
DXL	DOM	DXL	DOM	DXL	DOM
10	20	9.89	20.12	99.12	100.85
10	20	9.86	20.36	98.84	101.82
10	20	9.98	20.26	99.83	101.3
10	20	10.11	20.03	101.18	100.16
10	20	10.07	19.49	100.75	99.41
10	20	10.15	19.88	101.54	100.66
MEAN		10.540	20.034	100.66	100.428
SD		0.008	0.115888	0.3437	1.093741
%RSD		1.628	1.154952	1.718159	1.08908

Table 5: Results of Repeatability Precision of DXL

Sr. No	Peak Area	
	DXL (20µg/ml)	DOM (20µg/ml)
1	9508253	3090066
2	9518253	3190066
3	9528253	3180066
4	9527253	3050066
5	9518263	3160066
6	9526253	3070066
Mean	9521088	3123399
±SD	7704.242	60553.01
%RSD	0.080918	1.938689

Table 6: Results of Accuracy for DXL and DOM by RP-HPLC Method

Level of % Recovery	Sr. No	Amount of Standard Drug Added (µg/ml)		Total Amount Found (µg/ml)		Total Amount Recovered (µg/ml)		%Recovery	
		DXL	DOM	DXL	DOM	DXL	DOM	DXL	DOM
80%	1	48	24	108.54	54.31	48.54	24.31	101.1	101.2
	2	48	24	107.89	53.69	47.89	23.69	99.77	98.7
	3	48	24	108.08	54.13	48.08	24.13	100.1	100.54
100%	1	60	30	120.34	60.21	60.34	30.21	100.5	100.7
	2	60	30	121.15	60.48	61.15	30.48	101.9	101.6
	3	60	30	119.86	59.94	59.86	29.94	99.76	99.8
120%	1	72	36	132.62	66.34	72.62	36.34	100.8	100.9
	2	72	36	132.04	66.14	72.04	36.14	100.5	100.3
	3	72	36	132.35	66.46	72.35	36.46	100.4	101.2

Table 7: Statistical Validation Data for Accuracy Study of DXL and DOM

Level of % Recovery	Mean* (% Recovery)		± SD		% RSD	
	DXL	DOM	DXL	DOM	DXL	DOM
80%	100.32	100.14	0.489	0.916	0.488	0.916
100%	100.72	100.7	0.768	0.636	0.763	0.631
120%	100.56	100.8	0.147	0.324	0.146	0.321

Conclusion

A simple, accurate, precise, reproducible, and cost-effective RP-HPLC method was successfully developed and validated for the simultaneous estimation of Dexlansoprazole and Domperidone in pharmaceutical dosage forms. The method was validated in accordance with ICH guidelines and demonstrated satisfactory results for parameters including linearity, assay, accuracy, precision, limit of detection, and limit of quantitation. Owing to its reliability and reproducibility, the developed method can be effectively employed for routine quality control analysis of these drugs in pharmaceutical formulations.

References

- Nithyasri, Kabra P, Vijaya BS, Vijayantimala P, Sachindra LN, Giri A. Development and validation of UV spectrophotometric methods for simultaneous estimation of Dexlansoprazole and Domperidone in pharmaceutical dosage form. *World J pharma ceu life sci.*,2026;12(9):115-120.
- Sadafi S, Azizi A, Pasdar Y, Shakiba E, Darbandi M. Risk factors for gastroesophageal reflux disease: a population based study. *BMC Gastroentero.*,2024;24(64):2-7.
- Li Q, Duan H, Wang Q, Dong P, Zhou X, Sun K, *et al.* Analysing the correlation between gastroesophageal reflux disease and anxiety and depression based on ordered logistic regression. *Sci Reports.*,2024;14(1):1-11.
- Sharma S, Goyal S, Chauhan K. A review on analytical method development and validation. *International Journal of Applied Pharmaceutics.*,2018;10(6):8-15.
- International council for harmonisation of technical requirements for pharmaceuticals for human use. Validation of Analytical Procedures [Internet]. ICH, 2023. [cited 2025 March15]. Available from https://database.ich.org/sites/default/files/ICH_Q2%28R2%29_Guideline_2023_1130.pdf
- Drugbank.com >drugs (<https://go.drugbank.com/drugs/DB05351>) (Accessed on 10-Jul-2025).
- Drugbank.com >drugs (<https://go.drugbank.com/drugs/DB01184>) (Accessed on 10-Jul-2025).
- Prasad KSA, Abhiram MS, Raju HV. Analytical method development and validation for simultaneous UV spectrophotometric estimation of Dexlansoprazole and Domperidone. *World J Pharm Res.*,2025;14(6):979-93.
- Konduri RB, Bhavani T, Srividya P, Eswaraiiah MC. Validated UV spectroscopic method for determination of domperidone in bulk and pharmaceutical formulation UV spectroscopic method. *Research Journal of Pharmacy and Technology.*,2025;18(1):317-20.
- Ambi A, Wakte P, Bhusari S. Development and Validation of UV-Visible Spectrophotometric Method for Estimation of Dexlansoprazole. *Journal of Drug Delivery & Therapeutics.*,2025;15(8):148.
- Bhole R, Bonde C, Ghansham G, Shailendra G. Stability indicating validated novel RP-HPLC method for Dexlansoprazole and LCMS/MS study of degradation product. *Pale Med Pharm J.*,2024;9(1):81-106.
- Kerthi LB, Deepthi R Rao SY, Rao VK. A Novel RP-HPLC method development and validation for the estimation of Dexlansoprazole in bulk and extended-release capsule. *World J Pharm Research.*,2021;10(8):1169-80.
- Kumar STS, Akkamma HG. Analytical method development and validation of Dexlansoprazole by RP-HPLC method in bulk and pharmaceutical dosage form. *J Eme Tech Inno Res.*,2021;8(6):882-9.