Research Article

UV Spectrophotometric Method for Estimation of Dextromethorphan in Bulk and Syrup Formulation by Area Under Curve Method

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ABSTRACT

A new, simple, rapid and novel spectrophotometric method has been developed for estimation of Dextromethorphan hydrobromide (DXM). DXM is antitussive drug. For this Area under Curve Method is used. The method involved measurement of AUC at wavelengths at 250 and 295 nm. The solutions of standard and the sample were prepared in water. Beer's law obeyed in concentration range of 10 to 50 µg/ mL and correlation coefficient 0.9995. These method was validated for precision, reproducibility, linearity and accuracy as per ICH guidelines. The method was found to be simple, accurate, precise and economical.

Keywords: Area under curve, Dextrometharphan hydrobromide, ICH guidelines.

1. INTRODUCTION

Dextromethorphan hydrobromide (DXM) is antitussive (cough suppressant) drug used for the pain relief and in psychological conditions. It acts on cough centre to elevate the threshold for coughing[1]. Chemically, it is morphinan, 3-methoxy-17-meth (9, 13, 14)-, hydrobromide. DEX is rapidly adsorbed from the gastro-intestinal tract. It is metabolized in the liver and excreted in the urine as unchanged DEX and demethylated metabolites including DEX, which has some cough suppressant activity.[2] Different methods have been reported for the determination of DEX in the bulk drug, in the dosage forms with other drugs in cough-cold products and in biological samples.

Literature survey revealed, different methods have been reported for the determination of DXM in bulk drug and in dosage forms in combination with other drugs. HPLC have been reported[3] the first and second-derivative technique UV spectrophotometry,[4] capillary electrophoresis,[5] gas chromatography ,[6-7] Liquid chromatography[8] and thin layer chromatography[9].

Among the various methods available for the determination of drugs, spectrophotometry continues to be very popular, because of their simplicity, specificity and low cost. This study presents new spectrophotometric method for the determination of dextromethorphan in syrup.

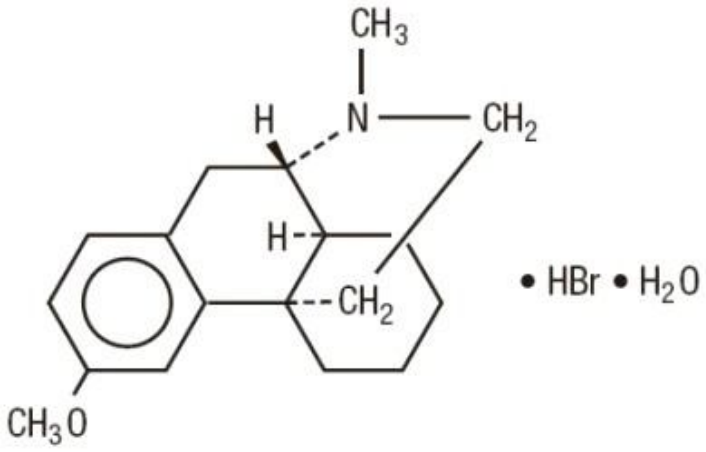

Fig. 1: Structure of Dextromethorphan HBr

2. MATERIAL AND METHODS

2.1 Instrumentation

Shimadzu UV 1800 double beam UV-visible spectrophotometer was used along with 1.0 cm path length matched pair of quartz cell for spectrophotometric method. Digital Balance: Shimadzu ATX224. Calibrated glassware was used for the study.

2.2 Reagents and chemicals

DXM reference standards was purchased from chemdyes chemicals Pvt. Ltd,. Analytical grade methanol was purchased from Suvidhanath laboratories Pvt Ltd. All the reagents were of analytical grade. Glass double distilled water was used throughout the experiment.

2.3 Preparation of standard stock solutions and calibration curve

Standard stock solution of pure drug containing 1000 µg/mL of DXM prepared in methanol. The working standard solutions of the drug were obtained by dilution of the stock solution in the distilled water. Series of solutions with conc. 10, 20, 30, 40, 50 µg/mL of DXM were used to prepare calibration curve. Solutions were scanned and proposed methods were applied. Water was used as a blank solution.

2.4 Preparation of sample stock solution

A requisite volume of the drug equivalent to 10 mg was transferred into a 100 mL volumetric flask (100 µg/mL). From this 2.5 ml was withdrawn and and diluted upto 10 ml.

2.5 Method: Area under curve (AUC)

It involves the calculation of integrated value of absorbance with respect to the wavelength between two selected wavelength. Area calculation processing item calculates the area bound by the curve and the horizontal axis. The horizontal axis is selected by entering the wavelength range over which the area has to be calculated. The wavelength range is selected on the basis of repeated observations so as to get the linearity between area under curve and concentration.

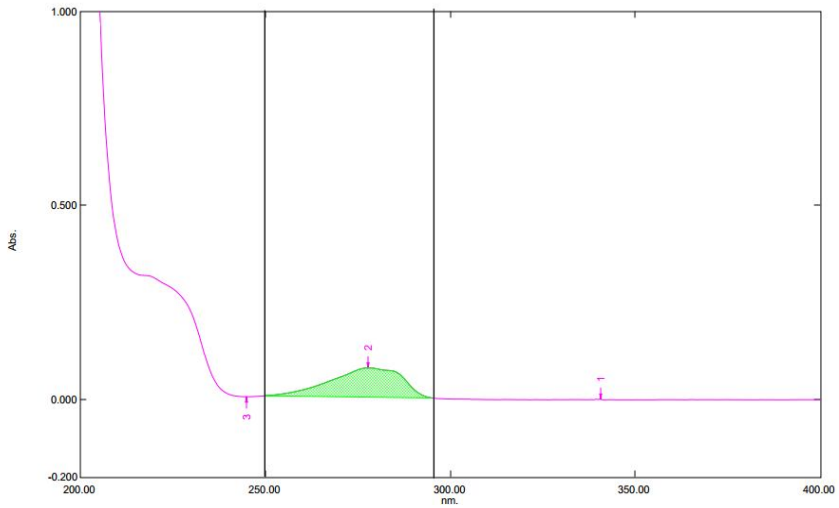

Fig. 2: Selection of AUC wavelength

2.5 a Preparation of calibration curve

Aliquots of working standard solution (1 – 5 ml) were transferred into a series of 10 ml volumetric flask, diluted up to mark with distilled water and scanned in the spectrum mode from the wavelength range 200-400 nm. A calibration curve was prepared by plotting the area versus concentration. The calibration curve was linear in concentration range of 10 – 50 µg/ml.

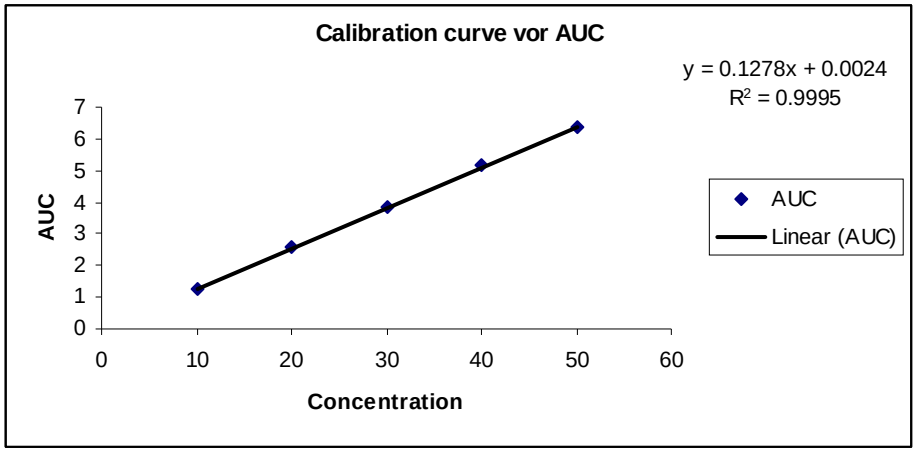

Fig. 3: Calibration curve for AUC

3. RESULTS AND DISCUSSION

3.1 Method validation

The Method was validated as per ICH guidelines using different parameter.

3.1.1 Linearity

The linearity was evaluated by analyzing different concentration of standard solution of DEX. The Beer Lambert's law was obeyed in the concentration range of 10-50 µg/ mL with regression coefficient of 0.9995

3.1.2 Ruggedness

Ruggedness of the proposed method is determined by analysis of aliquots from homogenous slot by two analyst using same operational and environmental conditions.

3.1.3 Accuracy (% recovery)

The accuracy of the methods was performed by calculating recovery of DEX by the standard addition method. Known amounts of standard solutions of DEX were added at 80%, 100% and 120% levels to pre quantified DEX sample solutions of 25 µg/ml. The amount of dextromethorphan was estimated by applying obtained values to the respective regression equations.

3.1.4 Precision

To determine the precision of the method, Dextromethorphan solutions at linear concentration were analyzed each three times. Solutions for the standard curves were prepared fresh everyday.

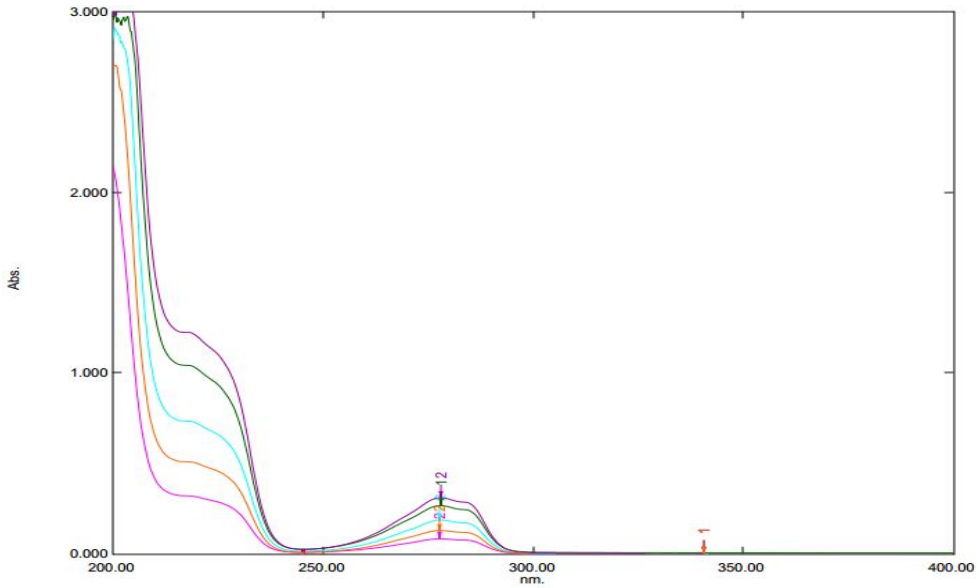

Fig. 4: Overlay spectra for Dextromethorphan

Table 1: Spectrophotometric characteristics and statistical data of the regression equations

Parameters	Results
λmax (nm)	250-295
Beer`s range (µg/ mL)	10-50
Regression equation	Y=0.1278X + 0.0024
Correlation coefficient	0.9995
Intercept	0.024
Slope	0.1278

Table 2: Results of Analysis of Syrup Formulation

S. No	Label claim	Amount in test solution	Amount found
Sample-1	10mg/5ml	25 µg/mL	24.92 µg/mL

Table 3: Intra and interday Precision

Label Claim	Amount in solution	Intraday Precision			Interday Precision
		Set 1	Set 2	Set 3	
10mg/5ml	25µg/mL	99.64%	99.64%	99.55%	99.51%

Table 4: Recovery data of Dextromethorphan

Level of % Recovery	Concentration Taken (µg/ mL)	Concentration estimated(µg/ mL)	% Analytical Recovery
80 %	33	32.97	99.90 %
100 %	35	34.94	99.82 %
120 %	37	36.73	99.27 %

CONCLUSION

No Area under curve spectrophotometric methods have been described for the determination of Dextromethorphan. The present study was undertaken with an objective of developing simple, sensitive and reliable analytical method like UV-Visible spectrophotometry for estimation of Dextromethorphan. The results of our study indicate that the proposed UV spectroscopic methods are simple, rapid, precise and accurate.

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