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Development and Validation of a UV Spectrophotometric Method for Analysis of Dexmedetomidine HCL

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ABSTRACT

A simple, accurate, rapid and reproducible UV-Spectroscopy method have been developed and validated for the estimation of Dexmedetomidine HCl in pure and dosage form. The absorbance of drug measured at 214nm wavelength in 0.9% NaCl solution. The range of linearity was found to be 2-10 µg/mL with the linearity equation of Y=0.0499x + 0.0517 and correlation coefficient is 0.9988. Developed method was validated according to the ICH Q2(R1) guidelines. The % RSD values for interday and intraday were found to be less than 2%. And % Recovery were in between 99.88-100.12%. LOD and LOQ were found to be 0.060 and 0.183 respectively. The result concluded that the developed method is accurate, precise and reproducible.

Key words: Dexmedetomidine HCl, 0.9% NaCl, UV Spectrophotometric method.

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INTRODUCTION

Dexmedetomidine HCl is chemically 4-[(1S)-1-(2,3-dimethylphenyl)ethyl]-1H-imidazole monohydrochloride with $C_{13}H_{16}N_2$.HCl chemical formula and 236.74048 gm/Mol molecular weight.[1]The Dexmedetomidine Hydrochloride is use for sedation of initially incubated and mechanically ventilated patients during treatment in an ICU, and sedation of non-intubated patients prior to or during surgical and other procedures.[2]Dexmedetomidine HCl API is official in USP 37, this monograph is in draft.[10]However, literature survey reveals that the many HPLC[3-8] methods in biological fluids has been reported, but there is no method is reported for the estimation of Dexmedetomidine HCl by UV Spectrophotometric method. So the aim of present research work is to development and validation of a UV Spectrophotometric method for analysis of Dexmedetomidine HCl. The chemical structure of drug were shown in figure 1[1].

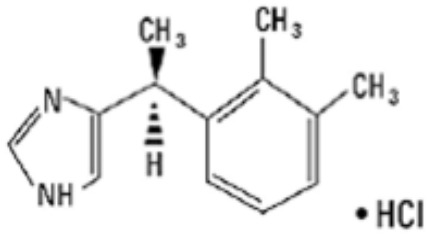

Figure 1: Chemical structure of Dexmedetomidine HCl.

MATERIALS AND METHOD

Instrument

Double beam UV-visible spectrophotometer SHIMADZU (MODEL 1800) with 1 cm matched quartz cuvettes, and software is UV- Probe Version 2.31. All weights are measured on electronic analytical balance (Wensar Dab 220).

Reagents and Materials

Dexmedetomidine HCl Injection were kindly gifted by reputed pharmaceutical company in Ahmedabad, India. Distilled water and Whatmann filter paper were used during the whole research work.

Selection of Diluent

0.9 % NaCl solution used as diluent, drug shows good solubility in 0.9 % NaCl.

Preparation of Standard Solutions

Preparation of standard solution of Dexmedetomidine HCl

Transfer an accurately weighed quantity about 5.0 mg of Dexmedetomidine HCl working standard in to 50 mL volumetric flask. Add 25 mL of diluent (0.9 % NaCl) and dissolve it. Dilute

to volume with diluent and mix well (100 ppm). Dilute 2.0 mL& 2.5 mL of this (100 ppm) solution to 25 mL with diluent and mix well (8 ppm) (10 ppm).

Test preparation

Dexmedetomidine HCl injection as such 100 ppm. From that take0.5 mL, 1 mL, 1.5 mL, 2.0 mL, and 2.5 mL and dilute up to 25 mL with diluent (0.9 % NaCl) and mix well to produce concentration range 2, 4, 6, 8, 10 ppm respectively for Dexmedetomidine HCl.

Procedure

Measure the absorbance of test preparation at maxima 214 nm using UV spectrophotometer, use diluent as blank. Calculate the quantity of Dexmedetomidine HCl in percentage from the value of absorbance of standard preparation, test preparation and percentage potency of working standard used.

Method Validation[9]

The method was validated as per ICH Q2 (R1) guidelines.

Linearity (n=5)

The linearity of the method is its ability to bring out the test results which are directly proportional to the concentration of an analyte present in the sample. Dexmedetomidine HCl was linear with the range of 2-10 ppm concentration at 214nm. Plot the calibration curve of absorbance Vs concentration and find out $R^{2.}$

Precision (Repeatability) (n=6)

Precision was performed for 6 times with the same concentration (8 ppm).

Intermediate precision (Reproducibility) (n=3)

In intra day study of drug were performed on the same day at the interval of 2 hours for 3 concentrations (2, 6, and 10 ppm). In inter day study of drug performed on 3 different days with 3 concentrations (2, 6, and 10 ppm).

Accuracy (Recovery study) (n=5)

To check the accuracy of the proposed methods, recovery studies is carried out at 80%, 100%, and 120% of the test concentration as indicated by the ICH Guideline.

limit of detection and Limit of quantification

LOD and LOQ were determined from the linearity data.

$LOD = 3.3 \times \sigma/S$

$LOQ = 10 \times \sigma/S$

Where, σ = the standard deviation of the Intercept of the 5 calibration curve and S = slope of the calibration curve.

RESULTS AND DISCUSSION

This method shows absorption maxima at 214 nm (Figure: 2) and obeyed Beer's law in concentration range of 2-10 µg/mL (Figure: 4). The calibration curve is linear for the concentration range of 2-10 µg/mL (Figure: 4). The r^2 was 0.9988. The method was found to be precise and the % RSD values for interday and intraday were found to be less than 2% (Table: 3). The % Recovery were in between 99.88-100.12 % (Table: 4). The LOD and LOQ were found to be 0.060 and 0.183 respectively (Table: 5).

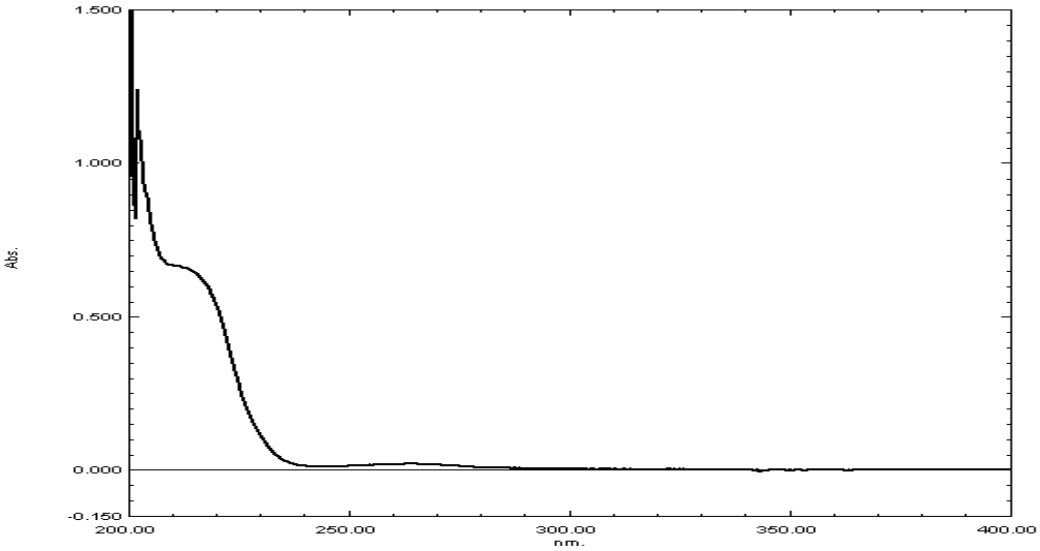

Figure 2: UV spectrum of Dexmedetomidine HCl Standard (0.9 % NaCl)

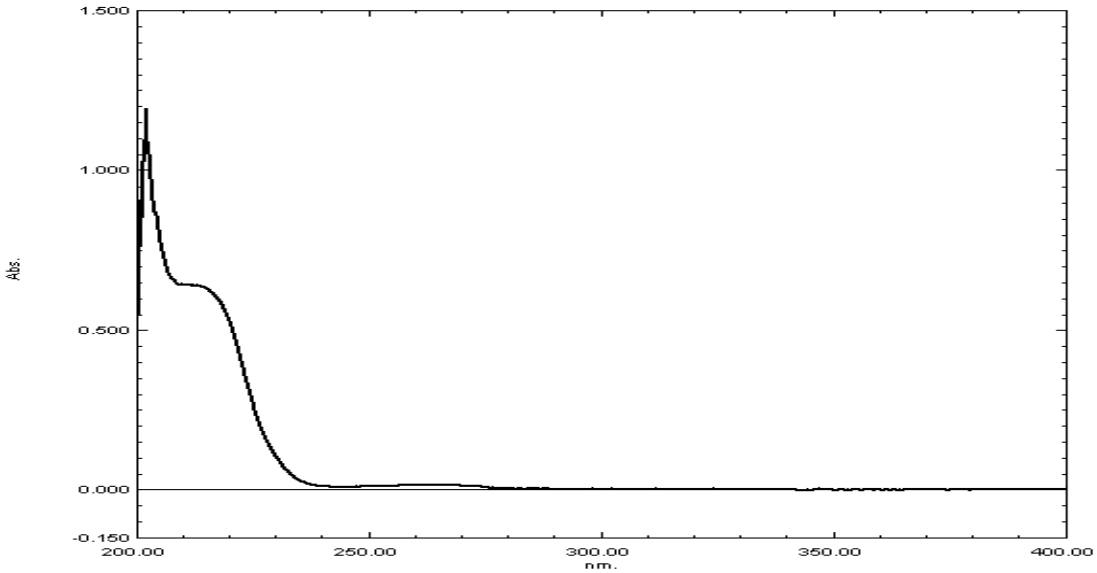

Figure 3: UV spectrum of Dexmedetomidine HCl test (0.9 % NaCl)

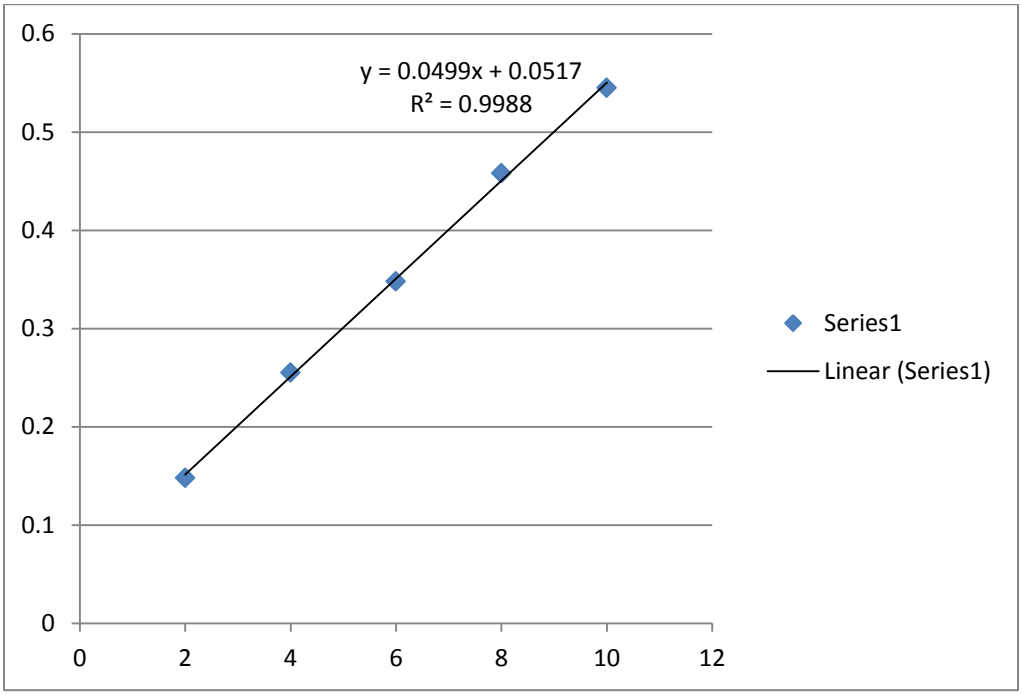

Figure 4: Linearity Graph for Dexmedetomidine HCl

Table 1: linearity data of Dexmedetomidine HCl (214 nm)

DexmedetomidineHCl (214 nm)		
Conc (µg/ml)	**Mean absorbance±SD**	**% RSD**
2	0.148±0.000471	0.31
4	0.255±0.000471	0.18
6	0.348±0.000471	0.13
8	0.458±0.000816	0.17
10	0.545±0.000471	0.08

Table 2: Repeatability data of Dexmedetomidine HCl (214 nm)

Dexmedetomidine HCl (214 nm)		
Conc (µg/ml)	**Mean absorbance±SD**	**% RSD**
6	0.347±0.000471	0.14

Table 3: Precision Data of Dexmedetomidine HCl (214 nm)

Parameters	Concentration (µg/ml)	Mean±S.D	% R.S.D
Intraday	2	0.147±0.000816	0.55
	6	0.347±0.000471	0.13
	10	0.544±0.000471	0.08
Interday	2	0.1463±0.001247	0.85
	6	0.346±0.001633	0.47
	10	0.5433±0.001247	0.23

Table 4: Recovery data of Dexmedetomidine HCl (214 nm)

Level	Concentration (µg/ml)	Total amount found (µg/ml)	% Recovery	SD
80 %	7.2	7.195	99.93	0.532
100 %	8	8.01	100.12	0.47
120 %	8.8	8.79	99.88	0.17

Table 5: Regression Analysis Data and Summary of Validation Parameter

Parameters	Dexmedetomidine HCl
Analytical Wavelength	214nm
Linearity range(µg/mL)	2-10
Regression Equation	Y= 0.0499x + 0.0517
Correlation coefficient (r^2)	0.9988
Standard deviation of the Y- intercepts of the 5 calibration curves	0.000899
Mean slope of the calibration curves	0.0491
Repeatability (RSD, n=6) %	0.14
Intraday Precision	0.147-0.544
Interday precision	0.1463-0.5433
LOD (µg/mL)	0.060
LOQ (µg/mL)	0.183

CONCLUSION

From the result it can be conclude that the method is a simple, accurate, precise, rapid and reproducible UV-Spectroscopy method for the estimation of Dexmedetomidine HCl in pure and dosage form. The % RSD of all parameters was found to be less than 2 %. So this method can use for routine analysis of Dexmedetomidine HCl in pure and dosage form.

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