ASIAN JOURNAL OF PHARMACEUTICAL AND CLINICAL RESEARCH

INNOVARE ACADEMIC SCIENCES Knowledge to Innovation

Vol 6, Suppl 4, 2013

ISSN - 0974-2441

Research Article

DEVELOPMENT AND VALIDATION OF SPECTROPHOTOMETRIC METHOD FOR THE ESTIMATION OF MOSAPRIDE IN BULK AND PHARMACUTICAL FORMULATION

P.D.S.SANKAR*, K.SUJANA[1], D.NOEL PREETHAM[2]

University College of Pharmaceutical Sciences, Acharya Nagarjuna University, Guntur-Email: pdssankar1@gmail.com

Received: 14 July 2013, Revised and Accepted: 8 August 2013

ABSTRACT

To develop simple, economical, precise UV methods for the estimation of Mosapride in bulk and pharmaceutical formulations. The methods are based on zero and first order spectroscopic techniques. Mosapride shows the maximum absorbance at 264nm (Method A), and in first order derivative spectra showed zero crossing point at 264nm with a sharp peak at 254nm when Δλ =10 (Method B). Drug followed the linearity in the range of 5-30µg/ml for both methods with correlation coefficient (r2) of 0.999. The results of analysis have been validated statistically and recovery studies confirmed the accuracy of the proposed method. The method was validated as per the International Conference on Harmonization (ICH) guidelines. The proposed method is recommended for routine analysis since it is rapid, simple, accurate and sensitive.

Keywords: Mosapride citrate, UV spectrophotometry, Derivative spectra.

INTRODUCTION

Mosapride (MO), is chemically known as 4-amino-5-chloro-2-ethoxy-N-[[4-[-[(4-fluorophenyl) methyl]-2-morpholinyl]-benzamide citrate dihydrate. It is listed in Martindale-the complete drug reference [1]. It is used in reflux esophagitis and to enhance gastric motility [2-3].This drug is selective $5HT_4$ agonist [4]. Literature survey reveals that there is a few analytical methods reported in HPLC [5], visible-spectrophotometry [6, 7] and by using LC-MS in biological samples, only one UV spectrophotometric method [8] was reported so far. In the present investigation two simple and sensitive UV spectrophotometric methods have been developed for the quantitative estimation of Mosapride in bulk and its marketed formulations with good accuracy and economy. The structure of Mosapride is shown in (Figure 1)

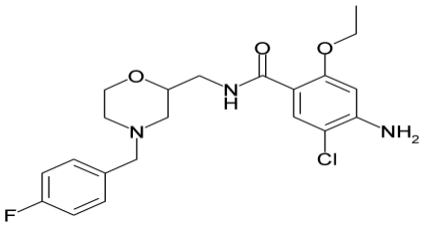

Fig1: Structure of Mosapride

EXPERIMENTAL WORK

All the chemicals used during the experimental work are of Analytical grade. A Shimadzu UV-1800 UV/VIS Spectrophotometer was used with 1cm matched quartz cells. Tablets of 5mg were procured from local market.

Preparation of standard solution

The pure drug of about 10 mg was weighed and transferred in to a 10ml volumetric flask. The drug was dissolved completely in a few ml of Methanol and made up to the final volume with double distilled water to get a stock solution of concentration 1000µg/ml. Aliquots of standard stock solution were pipette out and diluted suitably with water to get the final concentration of standard solutions.

Zero order spectroscopic method

The solutions were scanned in the range of 400-200 nm (method A), against water as reference, and the peaks were observed in the spectra at 264nm and 309nm respectively. The wavelength selected for analysis of drug was 264 nm (Figure 2). The drug obeys the lamberts law in the range of 5-30 µg/ml. By using linearity plot ((Figure 3a) the quantification was carried out.

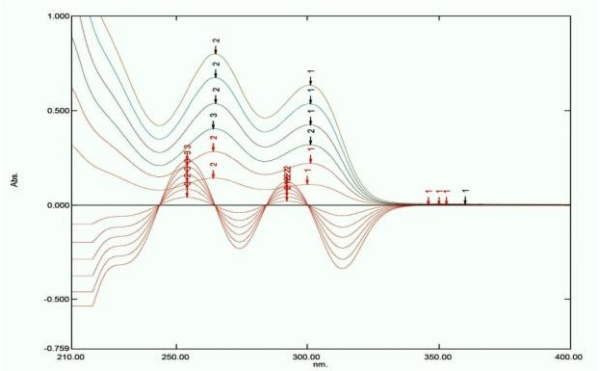

Fig.-2: spectra of linearity concentrations showing λ_{max} in zero order and 1st order

First order derivative spectroscopic method [9, 10]:

The first order derivative spectra (Method B), showed a sharp peak at 254 nm ((Figure 2). The absorbance difference at n=1 (dA/dλ) is calculated with Δλ=10 and scaling factor of 20 by inbuilt software of the instrument which was directly proportional to the concentration of standard solution. The standard drug solution was diluted so as to get the final concentration range of 5-30 µg/ml and calibration curve was constructed for dA/dλ against concentration of drug ((Figure 3b)

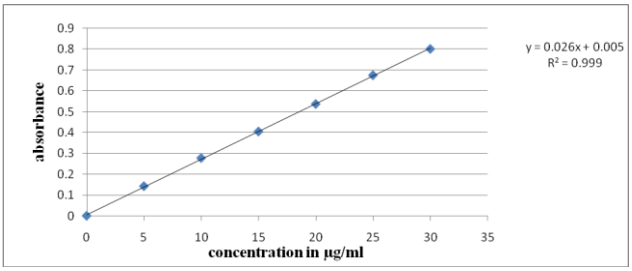

Fig3: (a)

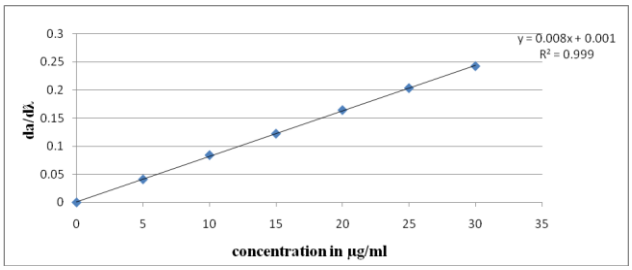

Fig.3: (b)

Fig.3: calibration curve, 3(a) for zero order, 3(b) for first derivative

Optical characteristics

Optical characteristics such as Beer's law limit (µg/mL), Correlation coefficient, Regression equation, Slope (m), and Intercept (c) were calculated (Table-1).

Table1: Optical characteristics

Optical characteristics	Method A	Method B
Beer's law limit (µg/ml)	5-30	5-30
Correlation coefficient (r^2)	0.9998	0.9998
Regression equation	y=0.0266x+0.0058	y=0.0081x+0.0012
Slope (a)	0.0266	0.0081
Intercept (b)	0.0058	0.0012
LOD	0.0736µg/ml	0.236 µg/ml
LOQ	0.223µg/ml	0.716 µg/ml

Analysis of tablet formulation

For the estimation of Mosapride citrate in pharmaceutical formulation by above two methods, 10 tablets of MOZA-5 brand were weighed and triturated to a fine powder. Tablet powder equivalent to 10mg was weighed and transferred to 100ml volumetric flask and dissolved in few ml of methanol with the aid of ultra-sonication for 15min; this was filtered through whatman filter paper no. 41 to get the stock solution of 100 µg/ml various dilutions were prepared from tablet solution and analyzed for six times and the concentration for both the methods was calculated by using calibration curve.

VALIDATION OF ANALYTICAL METHOD

The analytical method was validated according to ICH validation parameters [11]

Linearity

Fresh aliquots were prepared from standard stock solution ranging from 5-35 µg/ml and the absorbance values of each concentration was recorded at 264nm for zero order and at 254nm for 1st derivative method using water as blank. The drug shows linearity between 5-30 µg/ml for both methods (Table-2)

Table2: linearity of Mosapride

S.No	Concentration in µg/ml	Absorbance	
		Method A	Method B
1	5	0.142	0.041
2	10	0.277	0.084
3	15	0.404	0.122
4	20	0.536	0.164
5	25	0.674	0.203
6	30	0.801	0.242

Precision

In intraday study, concentration of replicates of drug was calculated on the same day for three times. In interday study the concentration of drug were calculated on three successive days which expresses the laboratory variation in different days. In both intra and interday precision study for the methods %RSD was calculated

Accuracy

Accuracy of the developed method was confirmed by performing recovery studies at three different concentration ranges 80%, 100%, 120% each one in triplicate (Table-3). From the recovery studies it was clear that the method is very accurate for quantitative estimation of tablet as the statistical results were within the acceptance range.

Table3: Accuracy studies of Mosapride

Method	Amount of µg/ml		% of drug added	Amount recovered	%recovered	% RSD
	Tablet	Pure drug				
	15	12	80	26.90	99.63	
Method-A	15	15	100	29.62	98.73	1.05
	15	18	120	33.26	100.81	
	15	12	80	26.76	99.11	
Method-B	15	15	100	29.68	98.93	0.75
	15	18	120	33.12	100.3	

Where,

Method-A: Calibration curve method
Method-B: 1st order derivative method

Limit of Detection and Limit of Quantification

The limit of detection and limit of quantification of Mosapride citrate by proposed methods were determined using calibration graphs.LOQ and LOD were calculated as 10*SD/S and 3.3*SD/S, respectively, where S is the slope of the calibration curve and SD is the standard deviation of response of least concentration of calibration curve in three replicates.

Robustness

Robustness of the method was determined by carrying out the analysis at five different wavelengths (±0.5nm). The respective absorbance was noted and the result was indicated by % RSD.

Ruggedness

Ruggedness of the method was determined by carrying out the analysis by two different analysts and the respective absorbance was noted. The result was indicated by % RSD

RESULTS AND DISCUSSION

The developed methods were found to be precise as the %RSD values for intra-day and inter-day were found to be less than 2%. Good recoveries (98.73% to 100.8%) of the drug were obtained at each added concentration, which indicates that the methods were accurate. The LOD and LOQ were found to be in sub-microgram level, which indicates the sensitivity of the method. The method was also found to be robust and rugged as indicated by the %RSD values which are less than 2%. The results of assay show that the amount of drug was in good agreement with the label claim of the formulation as indicated by % recovery (99.5%).

CONCLUSION

The proposed methods are simple, sensitive, and cost-effective. Validated in terms of precision, linearity and accuracy. The results are reproducible, and can be used successfully for the estimation of Mosapride citrate in bulk and its pharmaceutical formulations.

ACKNOWLEDGEMENT

The author is thankful to Dr. A.Prameela Rani, and University College of pharmaceutical sciences, Acharya Nagarjuna University, Guntur for providing necessary laboratory facilities.

REFERENCES

1. Sean C Sweetman. Martindale-The complete drug reference.2002, 33rdedition:1237
2. S.Budavari,Ed.In The Merck Index,13th ed, Merck and co.Inc., Whitehouse station, New Jersy,2001,pp. 6306, 8182
3. H.D.Langtry and M.I. Wilde. Proton pump inhibitors, Drugs 56: 447-486(1998).
4. Carisson L, Amos JG, Anderso B., the Journal of Pharmacology and Experimental Therapeutics, 282, 1997, 220-227.
5. Krishnaiah Yellela S.R, Murthy Tatikonda, Sankar Dannanag G. and Satyanarayana Valiveti, The determination of Mosapride citrate in bulk drugs and pharmaceutical dosage form using HPLC, Ana.Sci.2002, 18, 1269
6. B.K.Prabhakar, Shobhanamanjunath and S.Appalaraju. Spectrophotometric determination of Mosapride. Journal of the Indian Council of Chemists, vol.20, No. 1, 2003, pp.42-45.
7. B.S.Kuchekar, Usha Adagale, M.Nagar and S.B.Bhise. Spectrophotometric method for the estimation of Mosapride in tablets. Indian journal of pharmaceutical sciences. January-February 2003:85-86.
8. Patil shamkantS, DhabalePandurang N, KuchekarBhanudas S. Development and statistical validation of Spectrophotometric method for the estimation of Mosapride in pharmaceutical formulation International Journal of PharmaTech Research.Vol.1, No.4, pp 1458-1461, Oct-Dec 2009.
9. Deepak Bageshwar, Avinash Pawar, Vineeta Khanvilkar, Vilasrao Kadam. Simultaneous determination of Pantoprazole sodium and Itopride hydrochloride in pharmaceutical dosage form by first order derivative uv spectrophotometry Asian Journal of Pharmaceutical and Clinical Research,Vol. 3, Issue 3, 2010:221-223.
10. Rathod DR, Dole MN and Sawant SD. Spectrophotometric determination of Glipizide in bulk and tablet dosage form by absorption maxima, first order derivative spectroscopy and area under the curve. Asian J Pharm Clin Res 2012; 5(Suppl 3): 102-104.
11. Validation of analytical procedure: methodology Q2B.ICH Harmonized Tripartite Guidelines, (1996), 1-8.