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Development and validation of spectrophotometric method for estimation of troxipide in tablet dosage form

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ABSTRACT

Two simple, precise and economical spectroscopic methods have been developed for the estimation of Troxipide in bulk and pharmaceutical formulation. Method A is first order derivative spectroscopy where drug showed absorbance maxima 247.58 nm. Amplitude difference (dA/dλ) was calculated and plotted against concentration and regression equation was calculated. Method B applied was AUC (Area under curve) in which area in the wavelength range of 255-265 nm was selected for analysis of Troxipide. Drug followed the Beer-Lambert's law in the concentration range of 10-50 µg/ml (r^2=0.999) in 0.1 N HCl for both the methods. The % assay of marketed formulation for first order derivative and area under curve method was found to be 98.45% and 99.32% respectively. The methods were validated with respect to linearity, precision and accuracy studies. Recovery studies for first order derivative and area under curve method was found to be satisfactory. The methods were found to be simple, precise and accurate and can be employed for routine quality control analysis of Troxipide in bulk as well as its dosage form.

KEYWORDS

Troxipide (TRX);
Spectroscopic method;
First order derivative;
Area under curve (AUC).

INTRODUCTION

Troxipide is a novel gastro protective agent with anti-ulcer, anti-inflammatory and mucus secreting properties. Troxipide is chemically known as 3,4,5-Trimethoxy-N-(piperidin-3-yl) (Figure 1) benzamide[1]. It neither inhibits acid secretion nor has acid neutralizing activity, but has been clinically proven to heal gastritis and gastric ulcers. It has been postulated that Troxipide's mucosal protective effect in gastric ulcer and gastritis is exerted via the inhibition of inflammatory responses and neutrophil-mediated mucosal injury. It promotes ulcer repair by increasing collagen regeneration of the ulcer base and causes healing of peptic ulcer[2-4]. It is also used in the treatment of gastro esophageal reflux disease. Literature survey revealed that Troxipide is estimated by High performance liquid chromatography[5-9]. To our knowledge first order derivative UV spectroscopic and AUC methods are not available for estimation of Troxipide in single component formulation. Hence, an attempt has been made to develop new UV methods for its estimation in pharmaceutical formulation with good accuracy, simplicity, precision and economy.

EXPERIMENTAL

Instrumentation

An UV-Visible double beam spectrophotometer (Varian Cary 100) with 10 mm matched quartz cells

was used. Electronic balance (Model Shimadzu AUW-220D) was used for weighing.

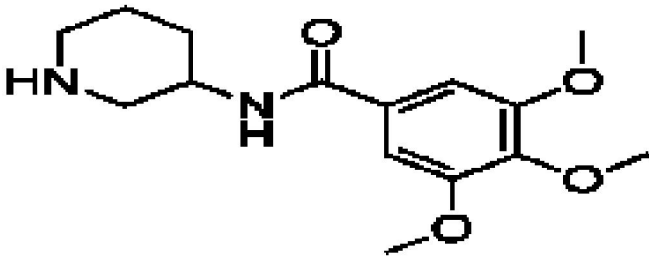

Figure 1 : Chemical structure of Troxipide molecule[9]

Reagents and chemicals

Pure Troxipide was obtained as a gift sample from Emcure Pharmaceuticals, Pune. Drug sample was used without further purification. TROXIPIDE tablets of 100 mg strength (Brand name-Troxpro) were procured from local pharmacy. Solvent used was 0.1 N HCl throughout the study.

Preparation of standard solution and calibration curve

Stock solution of drug having concentration 100 µg/ml was prepared by dissolving Troxipide in 0.1 N Hydrochloric acid. Aliquots of standard stock solution were pipette out and suitably diluted with 0.1 N HCl to get working standard solutions of analyte in the concentration range of 10-50 µg/ml of Troxipide and scanned in the range of 200-400 nm. For method A derivative amplitude of first derivative was measured at 247.58 nm and for method B area was integrated at wavelength range of 255-265 nm. Instrumental response and concentration obtained was used for construction of calibration curve. Beer's law was obeyed over the concentration range of 10-50 µg/ml by Troxipide.

Preparation of sample solution and formulation analysis

Twenty tablets were weighed and triturated to fine powder. Tablet powder equivalent to 10 mg of Troxipide was transferred to 100 ml volumetric flask, 80 ml of 0.1 N HCl was added to the same flask, sonicated for 5 min and filtered through What man filter paper No. 41 then diluted to 100 ml with 0.1 N HCl to get sample stock solution. Resulting solution was further diluted with 0.1 N HCl to obtain solution having concentration 30 µg/ml and proposed methods were followed to determine concentration of analyte and % assay was calculated.

METHODS

Method A: First order derivative spectroscopy

In the first order derivative method at absorbance difference n=1 showed a sharp peak at 247.58 nm (Figure 2). The absorbance difference at n=1 (dA/dλ) is calculated by the inbuilt software of the instrument which was directly proportional to the concentration of the standard solution. Dilutions of the analyte in the concentration range of 10-50 µg/ml were scanned in the first order derivative spectra. The calibration curve of dA/dλ against concentration of the drug showed linearity.

TRX = dA/dλ – intercept (C) / slope (m) (1)

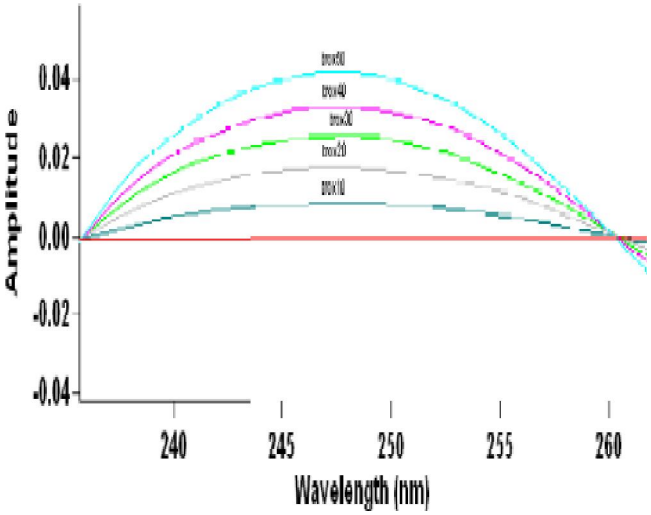

Figure 2 : First order derivative spectra of Troxipide in 0.1 N HCl (Con. Range 10-50 µg/ml)

Method B: Area under the curve method

The AUC method involves the calculation of integrated area of absorbance with respect to the wavelength between two selected wavelengths λ_1 and λ_2. For the selection of analytical wavelength, 30 µg/ml solution of TRX was prepared by appropriate dilution of standard stock solution and scanned in the spectrum mode from 200 to 400 nm. From the spectra of the drug, area under the curve in the range of 255-265 (Figure 3) was selected for the analysis. The calibration curve was prepared in the concentration range of 10-50 µg/ml at their respective AUC range. By using the

Full Paper

calibration curve, the concentration of the sample solution can be determined.

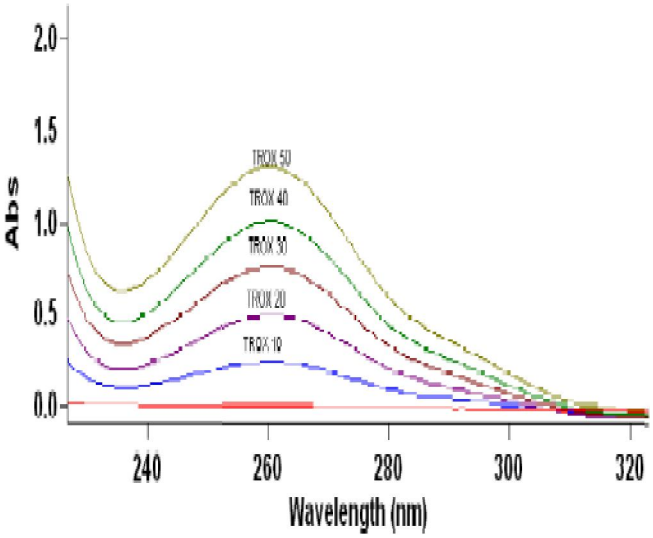

Figure 3 : Zero order spectra of Troxipide in 0.1N HCl (Conc. Range 10-50 µg/ml)

TABLE 1 : Optical characteristics and validation Data of Troxipide

Parameter	Method A	Method B
λ_{max} (nm)/wavelength range (nm)	247.58	255-265
Beer's- Lambert's range (µg/ml)	10-50	10-50
Coefficients of correlation (r^2)	0.999	0.999
Regression equation y = mx + c		
Slope (m)	0.00082	0.2603
Intercept (c)	0.000645	-0.36009
Precision (%RSD)		
Repeatability (n=6)	0.86	0.98
Intra-day (3×5 times)	1.73	1.54
Inter-day (3×5 days)	1.32	1.42
Analyst	0.92	0.78
Formulation Analysis (% Assay, % RSD)		
T1	98.45, 0.45	99.32, 0.39

RSD = Relative standard deviation, y= mx + c, where y is the absorbance and x is the concentration in µg/ml

Recovery studies

The accuracy of the proposed method was checked by recovery studies, by addition of standard drug solution to preanalysed sample solution at three different concentration levels (50%, 100%, and 150%) within the range of linearity for the Troxipide. The basic concentration level of sample solution selected for spiking of the drug standard solution was 30µg/ml of TRX.

Both the methods were validated according to ICH guidelines.

TABLE 2 : Recovery study of Troxipide for both methods

Formulation studied	Recovery level	Recovery of	Amount Spiked (µg/ml)	% Mean Recovery % RSD by n=3	
				Method A	Method B
Formulation I	50%	TRX	10	99.45,0.82	98.89,1.23
	100%	TRX	20	99.31,0.78	99.21,0.59
	150%	TRX	30	98.75,0.74	99.23,1.03

RESULT AND DISCUSSION

Under experimental condition described, calibration curve, assay of tablet and recovery study was performed. Various dilutions of standard stock solution were scanned separately. A critical evaluation of proposed method was performed by statistical analysis of data where slope, intercept, correlation coefficient are shown in TABLE 1. As per the ICH guidelines, the method validation parameters checked. Beer-Lambert's law was obeyed in the concentration range of 10-50 µg/ml for both the methods. Correlation coefficient was greater than 0.999 by both the methods. The proposed methods were also evaluated by the assay of commercially available tablet containing Troxipide. The results of formulation analysis are presented in TABLE 1. Recovery was found in the range of 98.75%-99.45 and 98.89- 99.23 by method A and B respectively (TABLE 2). The accuracy is evident from the data as results are close to 1005 and standard deviation is low.

CONCLUSION

The validated Spectrophotometric method employed here proved to be simple, economical, precise and accurate. Thus it can be used in IPQC test and for routine analysis of Troxipide in tablet dosage form.

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