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Research article

Zero order derivative spectroscopic estimation of olopatadine hydrochloride from eye drops

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Abstract

New simple, precise, accurate and cost effective UV spectrophotometric method was developed for the estimation of olopatadine hydrochloride from its ophthalmic dosage form by zero order (Dº) derivative spectroscopic method. The method utilized distilled water as a solvent. Zero order derivatives utilizes λmax of the drug as 299.50nm for analysis. The drug follows beer-lamberts law in the concentration range of 10-100µg/ml. The correlation coefficient of olopatadine hydrochloride is 0.999 and drug recovery 99.95% respectively. The developed UV spectrophotometric method was validated according to ICH guidelines with respect to accuracy, precision, ruggedness, specificity, LOD is 0.213 µg/ml and LOQ is 2.133 µg/ml. The proposed method can be implemented for routine analysis of olopatadine hydrochloride in its dosage forms.

Key words: Olopatadine hydrochloride, Zero order derivative, eye drops.

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1. Introduction

Olopatadine hydrochloride 11-((Z)-3-(Dimethyl-amino) propylidine)-6, 11-dihydrodibenz [b, e] oxepin-2-acetic acid, is a histamine H_1 receptor- antagonist and used as an Anti-Allergic and Anti-Inflammatory agent. It is a white crystalline powder, freely soluble in water and methanol.

Literature survey revealed that different methods were available for estimation of Olopatadine in human plasma using high performance thin layer chromatograph (HPTLC) [1], high performance liquid chromatography (HPLC), A radioimmunoassay (RIA) method is also available for determination of Olopatadine in plasma to support the pharmacokinetic studies in humans and some animals [2].

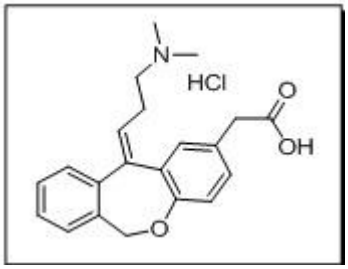

Figure 1. Structure of Olopatadine hydrochloride

Some reverse phase high performance liquid chromatography (RP-HPLC) methods were also reported for determination of

olopatadine hydrochloride in bulk drug and in pharmaceutical dosage form [3-5].

Some UV-spectrophotometric methods were also reported for estimation of olopatadine hydrochloride in eye drops by using HCl: methanol (50:50)[6], 50% aqueous methanol [7], 0.1N Sodium hydroxide, and 5mM ammonium formate [8] by taking absorption maxima at 206nm, 301nm, 220nm and 206nm respectively. But no method was available in distilled water. Volta metric method for determination of olopatadine hydrochloride in bulk drug and formulation was also reported [9]. Some simultaneous determination methods were also reported in water [10] and different solvents.

2. Materials and Methods

Chemicals and reagents

Active Pharmaceutical ingredient (API) i.e. Olopatadine Hydrochloride was obtained as a gift sample from Wockhardt pvt.ltd. Aurangabad, Maharashtra. The eye drop (OLOPAT, Ajanta pharma, Mumbai, India) was purchased from local market.

Instruments used

UV-Visible double beam spectrophotometer Shimadzu UV 1800, wavelength range 190-1100 nm band width 2nm, 1 cm quartz cells, slit width of 2 nm, instrument scan speed of 600 nm min-1 was used for analytical method development. The spectral data is processed by Shimadzu software UV Probe Ver.2.33. Shimadzu AX-200 electronic balance was used for weighing the drug.

Preparation of stock solution

A stock solution of Olopatadine hydrochloride was prepared, by accurately weighing 10 mg of drug and dissolving in separate 100 ml volumetric flasks and then diluted with distilled water up to the mark to get final concentration of 100 µg/ml. Further appropriate aliquots were pipette out from the standard stock solution into a series of 10 ml volumetric flasks, to get a set of dilutions of drug.

Preparation of sample solution

From the above stock solution 1ml-9ml was taken in different 10ml volumetric flasks and diluted to 10ml with the distilled water to produce the concentration of 10-90 µg/ml, respectively. All the drug solution was scanned (200-400 nm) against distilled water as blank. The absorbance maximum (λ max) was observed at 299.50 nm and the absorbances of a series of solution (10-90µg/ml) were recorded at the λmax. A graph was plotted by taking the concentration of the drug solutions on X-axis and absorbance on the Y-axis.

Assay of eye drops

The whole content of the eye drop was transferred in to the 50ml volumetric flask and volume was made with the distilled water. The solution was sonicated for 10 minutes. An appropriate dilution was prepared from this solution to get sample concentration (20 µg/ml) in the range of linearity for spectroscopic method.

The absorbance of sample solution was observed in multipoint calibration curve as a quantitative mode at 299.50 nm to get the concentration.

Validation of Method [11-14]

The validation of method was done by ICH guidelines for linearity, accuracy, precision (repeatability: within day, intermediate precision: different days), limit of detection (LOD), limit of quantitation (LOQ). The precision study

was done by recording the absorbance of the drug solution at (n=6) λ max and the % RSD was calculated. Accuracy was evaluated by the percent recovery studies for that the pre-analyzed formulation solution was spiked with 80%, 100% and 120% of pure sample drug and the % recovery was calculated.

3. Results and Discussion

Olopatadine hydrochloride is a white crystalline powder, freely soluble in water and methanol. Distilled water was preferred over methanol due to its cost effectiveness. The overlain spectra of olopatadine hydrochloride in distilled water at 299.50 nm were shown in Figure 2.

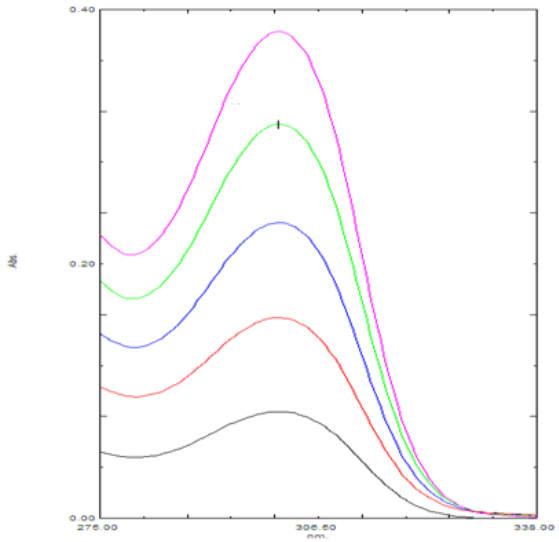

Figure 2. The overlain spectra of olopatadine hydrochloride

The absorbance's of different dilutions were measured as shown in table 1, and processed for the linear regression equation was found to be Y=0.006x+0.001 with correlation coefficient r^2= 0.999 as shown in figure 3. The Beer-Lambert's law limit was found to be 10-100 µg/ml.

Table 1. Calibration curve concentrations of olopatadine hydrochloride

Sr. No.	Concentration (in µg/ml)	Absorbance
1	10	0.07
2	20	0.14
3	30	0.21
4	40	0.28
5	50	0.34
6	60	0.41
7	70	0.49
8	80	0.56
9	90	0.62
10	100	0.69

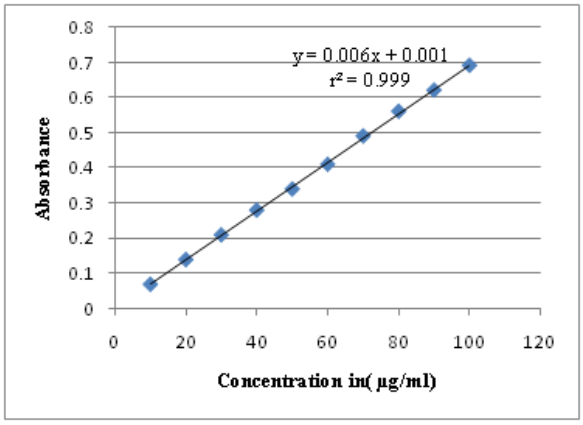

Figure 3. Calibration curve of olopatadine hydrochloride

The % RSD values in precision studies were found to be 0.0928 which is less than 2.0%. Indicating the method is more precise. The % recovery was found to be 99.95 %. The LOD and LOQ were found to be 0.213µg/ml and 2.1336µg/ml respectively. The characteristics of olopatadine were shown in table 2. The percentage of purity in the marketed formulation was found to be 98% ± 2%.

Table 2. Characteristics of Olopatadine hydrochloride

Sr. No.	Parameters	Values
1	λ max	299.50 nm
2	Linearity range	10-90 µg/ml
3	Slope	0.006
4	Intercept	0.001
5	Correlation coefficient	0.999
6	Accuracy (% Recovery)	99.75%
7	Precision (% RSD)	0.0928
8	LOD	0.213 µg/ml
9	LOQ	2.133 µg/ml

Conclusion

The validated spectrophotometric method developed is simple, rapid, accurate, precise, and cost effective and can be used for routine quality control analysis of the olopatadine in pharmaceutical formulations**.**

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