Available online at www.derpharmachemica.com

Scholars Research Library

Der Pharma Chemica, 2012, 4(6):2389-2392
(http://derpharmachemica.com/archive.html)

ISSN 0975-413X
CODEN (USA): PCHHAX

UV Spectrophotometric method for estimation of oxolamine citrate from bulk drug and pharmaceutical formulation

*** Rele Rajan V. and Sawant Swapnil A.**

Research Laboratory Department of Chemistry, D. G. Ruparel College, Dept. of Chemistry, Matunga, Mumbai-400 016.

ABSTRACT

A simple , rapid, sensitive and precise UV spectrophtometric method have been developed for the estimation of oxolamine citrate from bulk drug and pharmaceutical formulation. In this method oxolamine citrate showed maximum absorbance at about 237 nm in methanol. Beer's law was followed in the concentration range of 1 to 14 µg/ ml. Regression equation was found to be y = 0.066 x -0.0008 and coefficient of correlation was 0.9990. The proposed method is accurate, sensitive, reproducible and useful for the estimation of oxolamine citrate from bulk drug and pharmaceutical formulation.

Keywords: Oxolamine citrate, Methanol, UV-Visible spectrophotometer

---

INTRODUCTION

In this communication the present work proposes UV spectrophotometric method for assay of oxolamine citrate from bulk drug and pharmaceutical formulation. It's chemical name is 5- (2 -[diethyl amino] ethyl)3-phenyl-1,2,4 oxadiazole citrate. Oxolamine is an anti-inflammatory drug. This drug in Chemical Abstracts Service Registry Number [1]. Literature survey reveals no official method is reported for assay of this drug. A rapid , simple and reliable UV spectrophotometric method is developed for the determination of oxolamine citrate. This method can be used for the routine analysis and research organization. In the proposed work optimization and validation of this method are reported.

MATERIALS AND METHODS

Chemical and reagents:
Reference standard of oxolamine citrate was obtained from reputed firm with certificate analysis. A.R. grade methanol was used. All spectral absorbance measurements were made on Shimadzu UV-1800 with 10 mm matched cell.

Instrumentation:
Shimadzu 1800 was used with 10 mm matched quartz cell to measure absorbance of solution. A Shimadzu analytical balance with 0.01 mg was used.

Preparation of standard solution
Standard solution:
About 10 mg of standard oxolamine citrate was weighed accurately and transferred in 100 ml of volumetric flask. About 30 ml of A.R. grade methanol was added and sonicated for 15 minutes. The volume was adjusted up to the mark with methanol to give concentration as 100 µg /ml.

Experimental
Into a series of 10 ml graduated flask, varying amount of standard drug oxolamine citrate solutions were pipette out and volume was adjusted with methanol. Absorbance of the resulting solutions was measured at 237 nm using methanol as blank. (Fig. no. 1)

Estimation from tablets
Twenty tablets were weighed accurately and average weight of each tablet was determined. Powder equivalent to 10 mg of oxolamine citrate was weighed and transferred in 100 ml of volumetric flask. A 30 ml of methanol was added and sonicated for 15 minutes and filtered. The filtrate and washing were diluted up to the mark with methanol to give concentration as 100 µg /ml. Such solution was used for analysis.

Into series of 10 ml graduated flask, varying amount of sample solutions of oxolamine citrate were pipette out and volume was adjusted with methanol. Absorbance of the resulting solutions was measured at 237 nm using methanol as blank. The concentration of the drug in the given sample was calculated using calibration curve. The result of analysis are given in table no. 1

Validation
Accuracy:
Accuracy of the proposed methods was carried ascertained on the basis of recovery studies. It is performed by the standard addition method. Recovery studies were performed by adding standard drug at different levels to the pre-analyzed tablets powder solution and the proposed method was followed. From the amount of the drug estimated, the percentage recovery was calculated. The results of the analysis are shown in table no. 2

Precision:
The method precision was established by carrying out the analysis of homogenous powder blend of tablets. The assay was carried out of drug using proposed analytical method in six replicates. The value of relative standard derivation lie well within the limits indicated the sample repeatability of the method. The results obtained are tabulated in table no. 3

Inter-day and intra-day precision:
An accurately weighed quantity of tablets powder equivalent to 10 mg of oxolamine citrate was transferred to 100 ml of volumetric flask, sonicated for 15 minutes with methanol and diluted up to mark with methanol to get stock solution of concentration as 100 µg /ml.

The contents were filtered through whatmann filter paper no. 41. Aliquots portions were further diluted with methanol to get concentration of 10 µg /ml. of oxolamine citrate. The absorbance of final solutions was read after 0 hr., 3 hrs. and 6 hrs. in 10 mm cell at nm. Similarly the absorbance of the same solution was read on 1^{st}, 2^{nd} and 5^{th} day. The amount of oxolamine citrate was estimated by comparison with standard at 237 nm. The results are recorded are recorded in table no. 4.

RESULTS AND CONCLUSION

The proposed method was validated statistically and by recovery studies. The molar absorptivity and Sandell's sensitivity values show the sensitivity of methods while the precision was confirmed by the %RSD (relative standard deviation). Assay results of recovery studies are given in table no. 2. Results are in good agreement with labeled value. The reproducibility, repeatability and accuracy of this method was found to be good, which is evidenced by low standard deviation.

The proposed method is simple, sensitive, accurate, precise and reproducible. Hence it can be successfully applied for the routine estimation of oxolamine citrate in bulk and pharmaceutical formulation even at very low

concentration as 1 µg /ml. In conclusion the proposed method is simple, sensitive and accurate. It can be used for routine estimation of oxolamine citrate in bulk drug and pharmaceutical formulation.

Fig no. 1. UV spectrum of oxolamine citrate

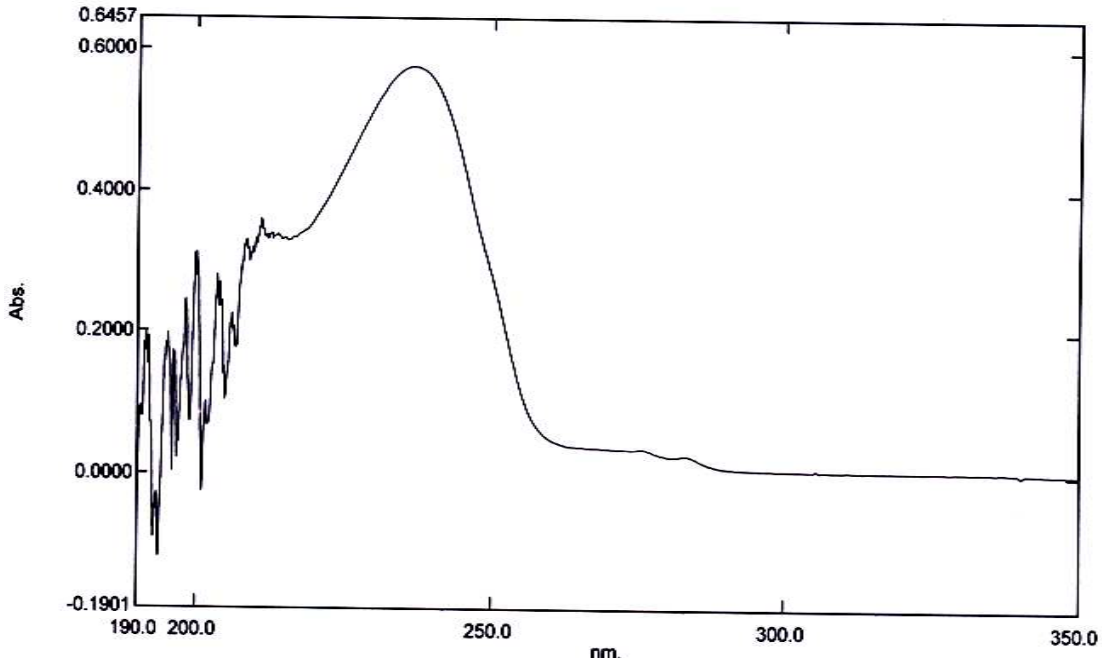

Table 1 : Optical and regression values of drug

Parameter	Values
λ max (nm)	237
Beer Law Limits (µg/ml)	1-14
Molar absorptivity(L/mol.cm)	28.71 x 10^3
Sandell's sensitivity	0.01515
Correlation coefficient(r^2)	0.9990
Regression equation (y=b+ac) Slope(a)	0.066
Intercept	0.00006

Table no. :2 Results of recovery of oxolamine citrate in bulk drug

Amount of sample added (µg/ml)	Amount of standard added (µg/ml)	Total amount recovered	Percentage recovery (%)	Standard deviation	Relative standard deviation (C.O.V.)
1	0	1.0064	100.64	0.01478	1.4692
1	1	2.0021	100.105	0.01607	0.8027
1	2	3.0041	100.136	0.01162	0.3069
1	3	3.984	99.600	0.02168	0.5443

Table no.3: Precision- method precision

Experiment no.	weight of oxolamine citrate taken in mg.	content in mg. of oxolamine citrate
1	10	10.064
2	10	10.0135
3	10	9.964
4	10	10.086
5	10	10.084
6	10	10.064
		Standard deviation= 0.04790 %RSD= 0.4768

Table no.: 4 Summary of validation parameter for intra-day and inter-day

Sr. no.	Parameters	Percentage
(A)	Intra-day precision (n=3) Amount found ± %RSD =	99.60 0.5443
(B)	Inter-day precision (n=3) Amount found ± %RSD	98.484 0.8882
(C)	Ruggedness Analyst to analyst (n=3) %RSD =	100.12 0.3845

Acknowledgements
Authors express sincere thanks to the Principal, Dr. Tushar M. Desai of D. G. Ruparel college.

REFERENCES

[1] Chemical Abstract Service Register Number **1949**-20-8.
[2] Ozan Pirol, Morat Sukuroglu and Tancel Ozden, *Journal of chemistry*, **2011**,8(3), 1275- 1279.
[3] Rele R.V., Patil S.P., *Research J. Pharm. And Tech*. **2011**,4(5), 787-789.