Available online www.jocpr.com

Journal of Chemical and Pharmaceutical Research, 2016, 8(7):108-112

Research Article

ISSN : 0975-7384
CODEN(USA) : JCPRC5

Dissolution Study of Oxolamine Citrate by UV Spectrophotometric Method in Pharmaceutical Dosage Form

Rele Rajan V.* and Sawant Swapnil A.

Central Research Laboratory, D. G. Ruparel College, Matunga, Mumbai 400016

ABSTRACT

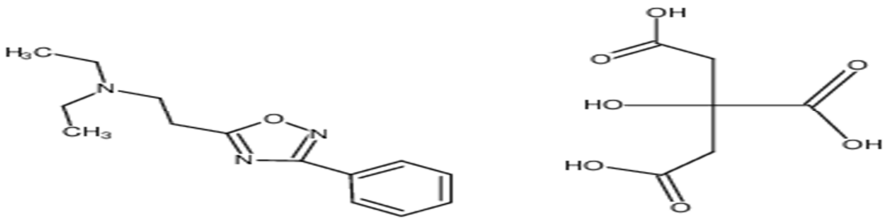
Dissolution study was carried for oxolamine citrate tablets containing 100 mg of active pharmaceutical ingredient. After the determination of solubility, the conditions selected were paddle at 75 and 100 rpm, with 1000 ml 0.01N HCl and phosphate buffer at 37°C ± 0.5°C. Under these conditions, the in vitro release profiles of oxolamine uncoated 100 mg tablets shown good results. The drug release was evaluated by UV spectrometric method at 237 nm in zero order spectrum. The linearity was found between 50- 150% with coefficient of co-relation was 0.9996. The standard devotions and relative standard deviation of precision were 0.015 and 0.015% respectively. The dissolution test developed and validated for oxolamine tablets was considered satisfactory.

Keywords: Oxolamine citrate, zero order spectroscopy, dissolution study, 0.01N HCl, 0.01M phosphate buffer.

INTRODUCTION

In this communication the present work proposes UV spectrophotometric method and dissolution study 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 is in Chemical Abstracts Service Registry Number [1]. Drug is not official in any pharmacopeia. Literature survey reveals liquid chromatography methods [2-5] and spectrophotometric [6-8] and non aqueous titration [9] for assay of oxolamine citrate. These methods can be used for the routine dissolution analysis in quality control laboratory. In the proposed work optimization and validation of these methods are reported. The structure of oxolamine citrate is shown in Fig.1.

Fig. 1: Chemical structure of oxolamine citrate



EXPERIMENTAL SECTION

Instrument and reagents

1) Shimadzu UV-spectrophotometer, model 1800 (Shimadzu, Japan) with spectral band width of 0.5 nm with automatic wavelength corrections by using a pair of 10 mm quartz cells. All spectral measurements were done by using UV-Probe 2.42 software.
2) Dissolution test was conducted using an Lab-India Disso 2000 dissolution tester using USP Apparatus at a temperature of 37°C ± 0.5°C.
3) Shimadzu analytical balance (0.01 mg) was used

Reagents and materials

1) Reference standard of oxolamine citrate was obtained from reputed firm with certificate analysis.
2) Millipore water was used for preparing dissolution media. All other reagents and Chemicals were of analytical or HPLC grade. Oxolamine citrate tablets containing Oxolamine citrate (100 mg) where procured from reputed firm.

Preparation of standard drug solution

Individual six tablets where weighed containing 100 mg oxolamine citrate and transferred into separate dissolution apparatus bowls containing 1000ml of 0.01N hydrochloric acid and 0.01 N phosphate buffer.

Dissolution study

The solubility study and percentage drug release were determined in 1000 ml of 0.01N hydrochloric acid and 0.01N Phosphate Buffer. Drug release tests were carried out with paddle method (USP apparatus II) at 75 and 100 rpm. The temperature of the cell was maintained at 37°C ± 0.5°C by using a thermostatic bath. Sampling aliquots of 5.0 ml were withdrawn at 15, 30, 45, 60, 75, 90 and 105 min and replaced with an equal volume of the fresh medium to maintain a constant total volume. After the end of each test time, sample aliquots were filtered and quantified.

The percentage content was calculated by validated UV- Spectrophotometric method and these contents results were used to calculate the percentage release on each time of dissolution profile. The cumulative percentage of drug released was plotted against time in order to obtain the release profile.

VALIDATION

Dissolution study

Linearity

To assess the linearity, 50 % - 150 % level of concentrated solutions were prepared. The calibration curve for oxolamine citrate was plotted. The results of the analysis are shown in table (1).

Table 1: Results of Linearity of oxolamine citrate for dissolution study

Parameters	Values
Correlation Coefficient (r)	0.9996
% Intercept (y)	0.003
Slope (m)	0.006

The accuracy was evaluated for the proposed method by adding known amount of Oxolamine citrate standard drug (80%, 100%, 120% level) to the tablet powder, which were subjected to dissolution test conditions described above. Each solution was analyzed in triplicate. The accuracy was calculated as the percentage of the drug recovered from the formulation matrix. The results of the analysis are shown in table (3).

Table 2: Results of recovery of oxolamine citrate for dissolution study

Amount of sample added in (mg)	Amount of standard added in(mg)	Total amount recovered in (mg)	Percentage recovery (%)	Standard deviation	Percentage of relative standard deviation (%) C.O.V.)
100	60	60.81	99.83	0.06	0.10
100	100	100.28	99.92	0.12	0.12
100	140	140.16	99.93	0.00	0.00

Precision

The methods precision was established by carrying out the analysis of tablets. The dissolution study was carried out in six replicates. The values of relative standard deviation lie well within the limits indicated the sample repeatability of the methods. The results obtained are tabulated in table 3.

Table 3: Precision- method precision

Experiment no.	Amount of oxolamine citrate taken in µg/ml.	%Assay
1	100	99.985
2	100	99.980
3	100	99.967
4	100	100.005
5	100	100.005
6	100	99.997
Standard deviation		0.015
%R.S.D.		0.015

Dissolution study

The solution stability was analyzed over a specified period of time, verifying the response of the sample solution stored at bench top condition (25°C). The spectrophotometric data obtained by the UV-spectrophotometric method from freshly prepared solution were compared with solution stability sample, table 8.

Table 8: Summary of validation parameter for solution stability

Time hrs	Mean % Assay
0.0	100.0419
12	99.56129
24	99.07234

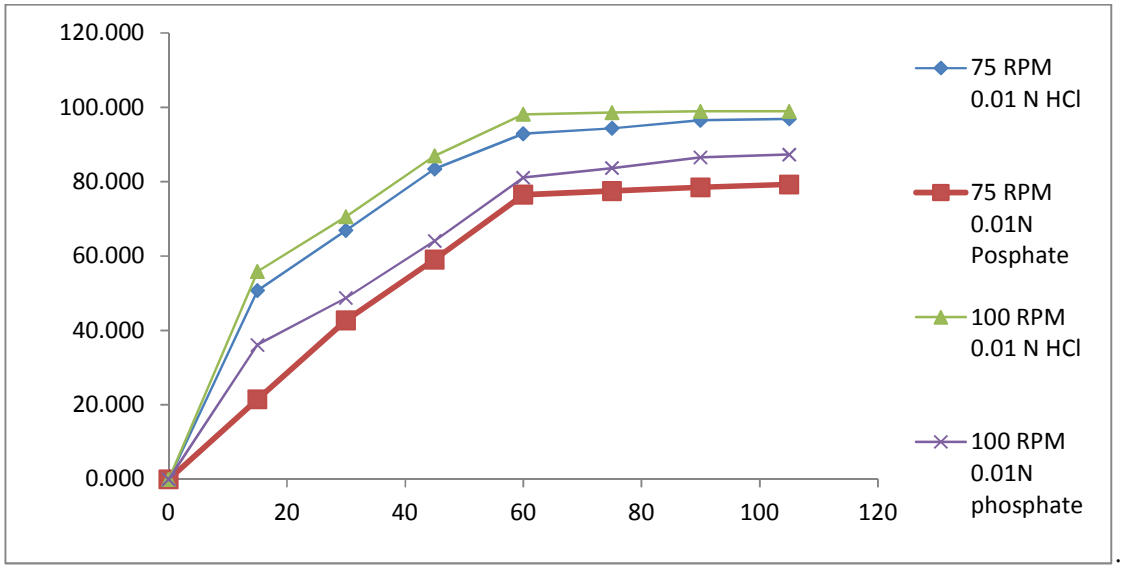
RESULTS AND DISCUSSION

When dissolution test is not defined in the monograph of the dosage form, comparison of drug dissolution profiles is recommended on three different dissolution media, in the pH range of 1-7.5. The selection of a dissolution medium may be based on the solubility data and dosage range of the drug product. Hydrochloric acid, phosphate buffer and purified water are typical mediums used for dissolution test and these mediums were evaluated. Oxolamine citrate was insoluble in alkaline medium and freely soluble in acidic and phosphate buffer medium. At 75 rpm, the cumulative percentage drug release was considerably less than that at 100 rpm in above said dissolution medium. It was observed that less than 50 %of drug was dissolved at 30 min in phosphate buffer at a speed of both 75 rpm and 100 rpm in 0.01N HCl medium showed 100 % cumulative drug release at 75rpm as well as 100 rpm level. Hence 0.01N HCl was used as dissolution medium for further study, table 9.

Table 9 : Summery of different revolution and disso medium

Time	75RPM		100RPM	
	0.01N HCl	0.01N Phosphate Buffer	0.01N HCl	0.01N Phosphate Buffer
0.0	0.000	0.000	0.000	0.000
15	50.742	21.491	55.850	36.117
30	66.958	42.732	70.615	48.760
45	83.454	59.059	86.970	64.111
60	92.943	76.560	98.163	81.109
75	94.395	77.509	98.637	83.677
90	96.516	78.513	98.944	86.552
105	96.907	79.267	98.972	87.305

Oxolamine citrate drug was completely released from its formulation at the end of the 90 min at a speed of 100 rpm. The cumulative percentages of drug in all the above said solutions were tabulated in Tables 9.



Dissolution profiles of oxolamine citrate in different dissolution medium at 75 rpm and 100 rpm.

Linearity of the method was evaluated at five concentration levels by injecting the standard solution in the range of 50–150 µg/ml. The calibration curve for oxolamine citrate was prepared by plotting the graph with area versus concentration. Calibration data for oxolamine citrate was shown in Table 1. The representative linear equation was y = 0.006x - 0.003 and correlation coefficient 0.999 for oxolamine citrate. Linearity observed in the expected concentration range demonstrated the suitability of the method for analysis. This indicated that the method is linear in the specified range for the analysis of oxolamine citrate in solid dosage form.

The recovery experiments were carried out by the standard addition method. The method was found to be accurate with % recovery of 99.83%–99.92% and has found with acceptable % RSD of not more than 2% at each level. The recoveries obtained by the dissolution method for oxolamine citrate were shown in Table 2.

The precision results of the dissolution method were evaluated by analyzing repeatability study (Table 3). The % RSD for repeatability study was observed at 0.015. The % RSD values not more than 2 % indicated the good precision of the method.

CONCLUSION

The dissolution test developed and validated for oxolamine tablets was considered satisfactory. The conditions that allowed the dissolution determination were 1000 ml of 0.1 % hydrochloride at 37°C ± 0.5°C, paddle apparatus, 100 rpm stirring speed. In these conditions, the oxolamine citrate was more stable. It can be concluded that the proposed method was fully validated and it was found to be simple, sensitive, accurate, precise, reproducible and relatively inexpensive and they gave an acceptable recovery of the analyte. Hence, the developed method can be recommended for routine quality control analysis of oxolamine citrate in tablet formulation.

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

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