Available online www.jocpr.com

Journal of Chemical and Pharmaceutical Research, 2014, 6(8):327-332

Research Article

ISSN : 0975-7384
CODEN(USA) : JCPRC5

Liquid chromatographic method for the determination of Triprolidine

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ABSTRACT

A simple, precise accurate and selective reversed phase high performance liquid chromatographic method for determination of triprolidine in preparations has been developed. In this method the active materials were separated using mobile phase consisting of ammonium acetate dissolved in 750 mL of deionized water and 4250 mL of absolute ethanol. The flow rate was 1.0 mL min^{-1} with UV detection at 232 nm. Chromatographic separation was achieved on ACE LC-Si (15.0 cm x 4.6mm, 5.0 µm) column. The method was found precise and linear over the concentration ranges of (0.057-17.058) mg/mL, with LOQ concentrations of (0.00208 mg/mL). Accuracy was found to be acceptable over levels 50 to 150 %. The results confirmed the selectivity of the analytical method.

Keywords: triprolidine, validation, HPLC.

INTRODUCTION

Triprolidine hydrochloride (Fig.1) is a pyridine derivative with the properties of antihistamine. It is a potent histamine H1-receptor antagonist (H1-blocker), with a rapid onset and long duration action, almost up to 12 hours. It is probably effective for the symptomatic treatment of seasonal and perennial allergic rhinitis, vasomotor rhinitis, and allergic conjunctivitis due to allergens, foods and prevention of allergic reactions to blood or plasma [1, 2].

The literature reveals that many works have been carried out for the determination of triprolidine in biological fluids as well as in formulations. This includes HPLC [3-6], electrophoresis [7], spectrophotometry [8-10].

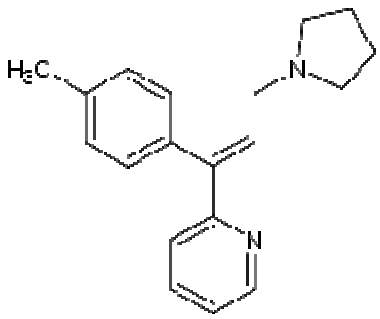


Fig.1: Structure of triprolidine

EXPERIMENTAL SECTION

Reagents and Sample

Triprolidine active materials used in the present work was from (USV Limited) pharmaceutical company. The chemicals were used for mobile phase preparations contain ammonium acetate anhydrous, hydrochloric acid and HPLC grade ethanol were purchased from Merck chemical company.

Instrumentation

The HPLC system used in this work consisted of a Simadzu LC-2010C HT Liquid chromatography (Japan), equipped with Photodiode array (PDA) SPD-M20A Detector, SIL-20AC Auto sampler and Software key version 1.21 SP1, copyright (c) 2002-2005 Computing integrator. Spectrophotometric measurements were carried out using Shimadzu UV-Visible-spectrophotometer model UV-1700, (Japan). The radiation degradation test was performed in Atlas XLS^+ sun test with source energy (765w/ m^2, xenon lamp (250nm - 765nm) and temperature 40 C°). Weighing of samples and standard at each solution preparation was performed on daily-calibrated balances of Mettler Toledo (AX26), minimum weight of (3.0 mg) on maximum tare of (2g), and Mettler Toledo (XS205), minimum weight of (20 mg) on maximum tare of (20 g).

Elma ultrasonic model S180H was used in standard and sample preparations Moreover, for filtration of mobile phase a vacuum pump vacuubrand GMBH membrane, model MZ2C also used, in addition to MicroSolv, syringe membrane filters 25 mm - 0.45 µm for filtration of standard and samples before injection in column.

Selection of wavelength

UV-VIS scan (200-400) nm was applied for solution of triprolidine HCl. A maximum absorbance was observed for each drug, 232 nm. This wavelength was selected for HPLC analysis, because it gives good responses linear, recovered and precise for the both active materials at the same time.

Preparation of mobile phase

Three grams of ammonium acetate were weighed and dissolved in 750 mL of deionized water, mixed with 4250 mL of absolute ethanol. The mixture was filtered through a 0.45 µm membrane filter and degassed for 10 minutes in ultrasonic prior to its use.

Preparation of standard solution

47.3 mg of triprolidine HCl from triprolidine $HCl.H_2O$ working standard were weighed and transferred completely to a 50 mL volumetric flask with the aid of about 30 mL of solvent (0.01 HCl), sonicated for 10 minutes with occasional shaking. The solution was diluted to volume with solvent and mixed well.

Preparation of samples solutions

A weight equivalent to five tablets was transferred completely to a 250 mL volumetric flask with the aid of about 150 mL of solvent. The solution was then shaked sonicated for 15 minutes, diluted to volume with solvent and filtered through a membrane filter.

Chromatographic conditions

Chromatographic separation was achieved on LC-Si (15.0 cm x 4.6mm, 5.0 µm) supplied by ACE company. The UV detector was operated at 232 nm wavelength. The mobile phase was pumped through the column at flow rate 1.0 mL/ min. Analyses were performed at ambient temperature and the injection volume was 20 µL

RESULTS AND DISCUSSION

The validation procedure was performed based on the ICH requirements. During the validation, the precision, linearity, limits of detection (LODs), quantitation (LOQs), accuracy, robustness, and specificity were investigated.

Precision

The precision of the method was established as repeatability, system and Intermediate precision. System precision was performed for six replicate injections of the same homogeneous standard solution prepared as described above. The method precision was assessed (performed) by analyzing six preparations of the drugs at the target concentration. Intermediate precision was established by performance the repeatability test on another day. The

acceptance criteria were set up as RSD value below 0.56 %, 0.66% and 0.83% respectively. The results of the method precision are presented in table 1 and pointed out that all criteria were fulfilled and the method is precise. As shown in figure 1.

Table (1): System, method and intermediate precision for triprolidine

System precision		Method precision(Day 1)	Intermediate precision(Day 2) (Ruggedness)
Injection #	Peak Area	% Assay	% Assay
1	3390847	100.2	99.5
2	3412628	99.4	99.9
3	3356385	100.7	99.0
4	3395246	100.0	99.5
5	3376524	101.1	101.4
6	3384621	99.5	99.6
Average	3386042	100.2	99.8
RSD %	0.56	0.66	0.83
Intermediate precision results from Day1 and Day 2			
Day 1		100.2	0.66
Day 2		99.8	0.83
Average		100.0	
RSD %		0.74	

Table (2) linearity for triprolidine

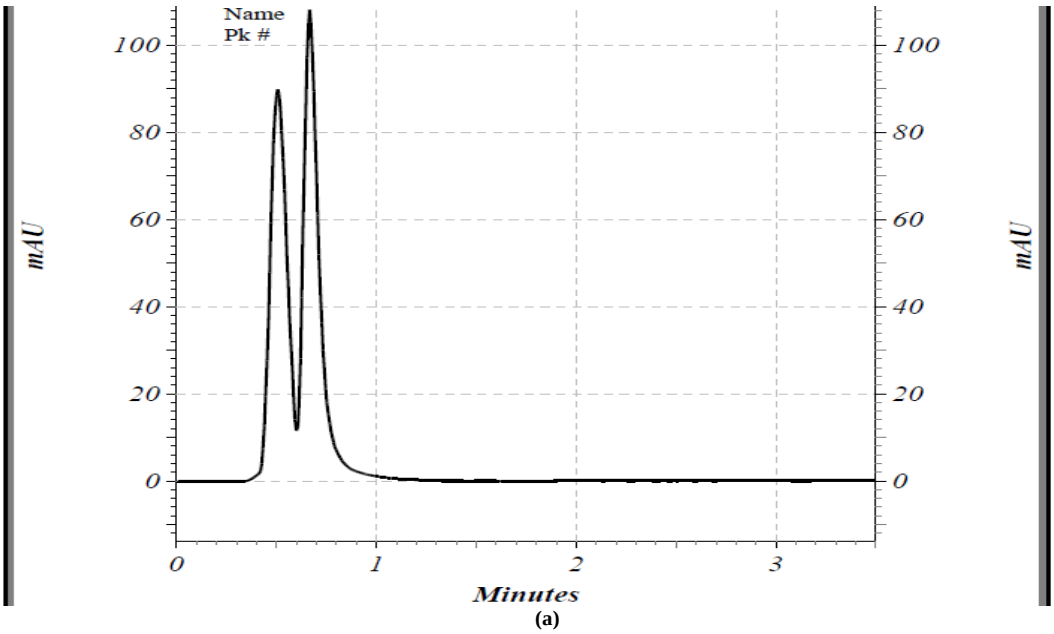
Level	concentration	Area
1	0.06	51576
2	0.70	670142
3	2.30	3356385
4	4.56	7347651
5	17.06	27565405

Table (3) spiking recovery for triprolidine

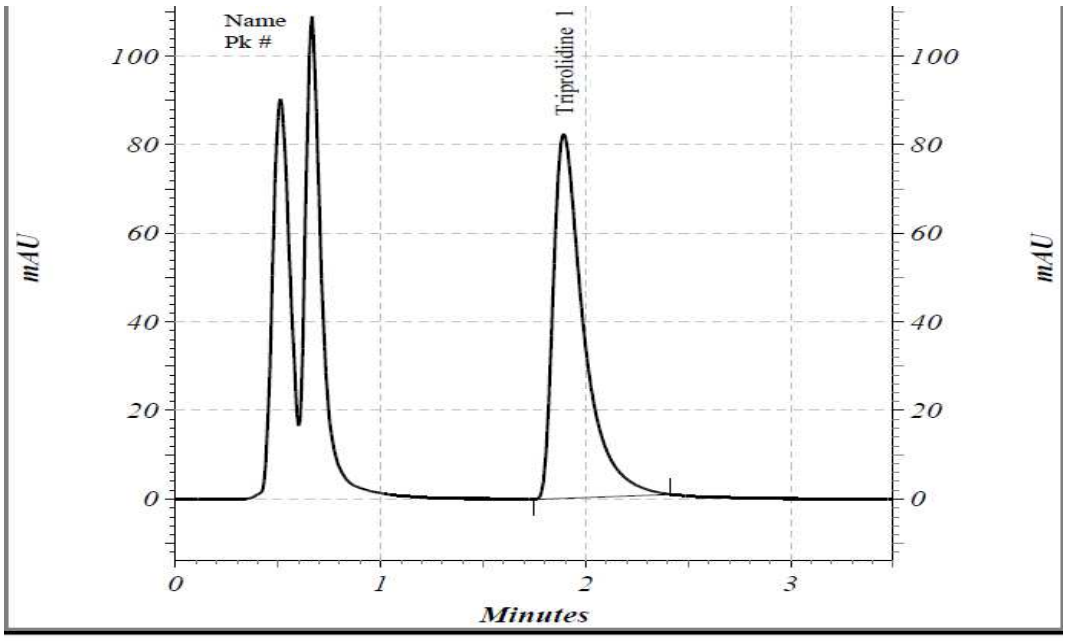
Levels	Preparation #	Average % Recovery
50 %	1	99.0
	2	99.8
	3	100.0
	Average	99.6
	%RSD	0.53
100 %	1	100.4
	2	99.4
	3	100.0
	Average	99.9
	%RSD	0.50
150 %	1	99.8
	2	99.9
	3	100.5
	Average	100.0
	%RSD	0.38

Linearity and Range

In order to evaluate the linearity of an assay procedure, a series of standards at different concentrations of the target concentration were prepared (0.057-17.058) mg/mL. After analyzing each preparation in duplicate, a linear regression analysis was performed on the average peak areas versus the concentrations of the levels versus the average concentrations of the levels preparations were studied Equation of calibration curve is (y = 1.63064e+006x - 231046). The present method was found linear over the concentration range of (0.06 - 17.1) mg/mL. The correlation coefficients was found to be (R^2 = 0.999767) as shown in figure 2 and table 2.

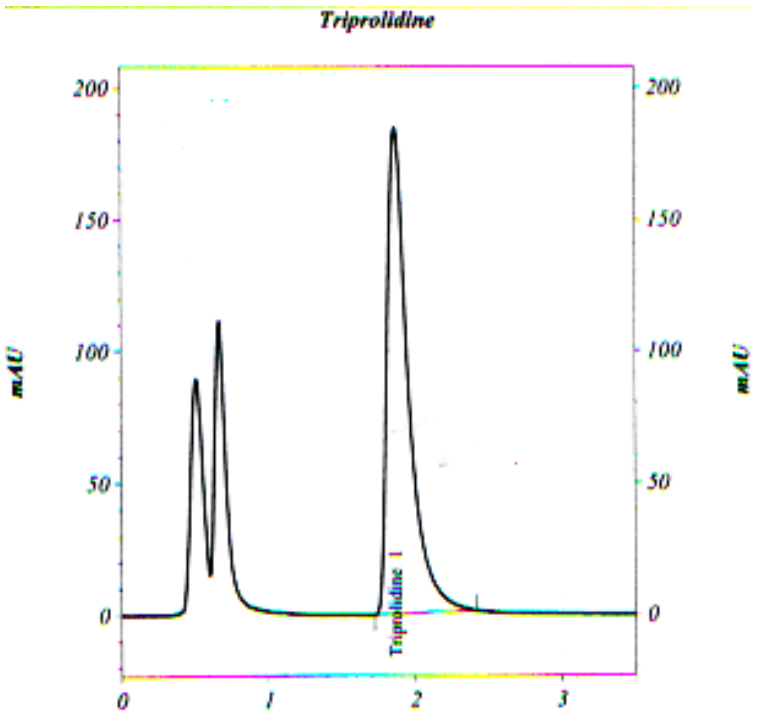


(a)



(b)

Figure (1): (a) A typical HPLC chromatogram injection of solvent (b) A typical HPLC chromatogram of (20 µL) injector standard solution



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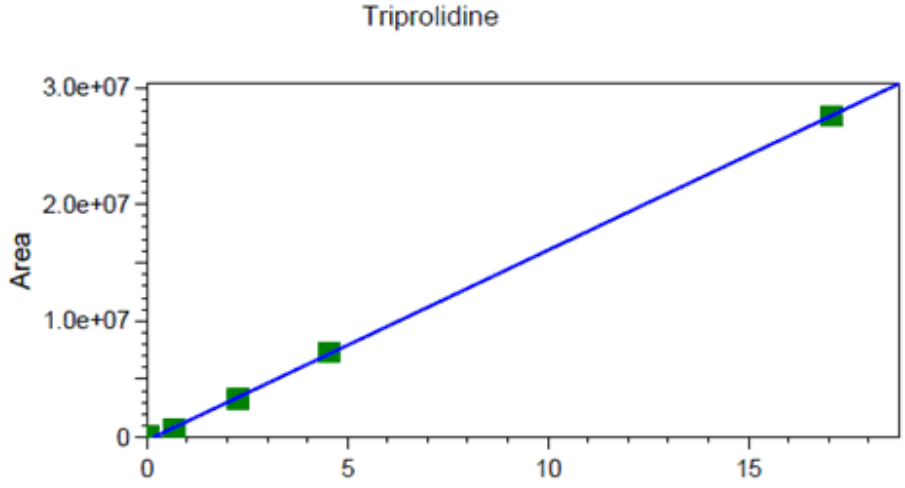

(b)

Figure (2): (a) A typical HPLC chromatogram of (20 µL) injector standard solution at concentration 4.6 for linearity taste. (b) Calibration curve for triprolidine

Accuracy (Recovery)

The accuracy was assessed by spiking recovery at three concentration levels, stock solutions of three different concentrations were prepared, (50 -150%). As shown in table (3), the obtained average percentage recoveries were within (99.0-100.0) of the actual spiked amount, which were acceptable. Which indicate no interference was obtained from excipients on the sample results; the closeness of the results to the true values.

Limits of detection and quantitation

The limit of detection (LOD) calculated as the concentration with respect to noise ratio, which generated a peak about 3 times as high as the noises height was found (0.000624 mg/mL), and the limit of quantitation (LOQ)

calculated as the concentration, which generated peak about 10 times as high as the noises height, was found (0.00208mg/mL).

Selectivity/ Specificity

Selectivity is a measure of the degree of interference (or absence thereof) in the analysis of complex sample mixtures. In the present work, the chromatograms of the samples were checked for the appearance of any extra peaks, injection of placebo, standard, no chromatographic interference from any of the excipients was found at the retention time of the examined active.

CONCLUSION

The present method has the advantages of simplicity, precision, accuracy and linearity. The relative standard deviation (RSD) for all parameters was found to be acceptable, which indicates the validity of method and assay results are within the limit. Hence, the proposed liquid chromatographic method can be used for routine analysis of active materials in quality control of the cited drugs in ordinary laboratories.

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