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Spectrophotometric determination of buclizine and pyrilamine in pharmaceutical formulations by Tpooo

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ABSTRACT

A simple, sensitive, selective and accurate spectrophotometric method Tpooo for the determination of Buclizine (BUCZ) and Pyrilamine (PYRA) in bulk drug and pharmaceutical formulations (tablets) has been described. This method is based on the extraction of drugs into organic layer of the dye Tpooo in presence of 0.1 N hydrochloric acid and the absorbances were measured at 480 nm. The results of analysis for this method have been validated statistically and by recovery studies.

KEYWORDS

Tpooo;
Spectrophotometric determination;
Buclizine;
Pyrilamine;
Statistical analysis;
Recovery studies.

INTRODUCTION

Tpooo (Tropaeolin ooo) generally used for the estimation of various drugs[1-3] Buclizine as hydrochloride (BUCZ) is a piperazine antihistamine (Figure 1) with antimuscarinic and central sedative properties. Chemically BUCZ is 1-[(4-chlorophenyl) Phenyl methyl] –4-[(4-(1,1 dimethyl ethyl) phenyl] methyl] piperzine; It is mainly used for the prevention of motion sickness when it should be given at least 30 minutes before traveling and it is also used in combination with analgesics to treat migraine attacks.

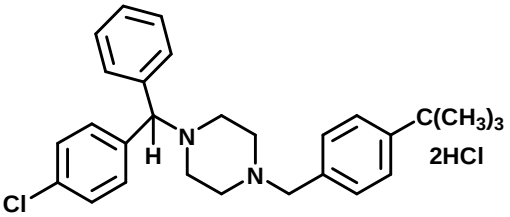

Figure 1: Buclizine

Pyrilamine[4] (as maleate PYRA, Figure 2)) is an antihistamine with a low incidence of side effects. It is effective for use in perennial and seasonal allergic rhinitis, vasomotor rhinitis, allergic conjunctivitis due to inherent allergens and foods, mild uncomplicated allergic skin manifestations of urticarea and angiodesma, angioedema, demo graphism and aneceoratum of reactions of blood or plasma. It is an antagonizing agent that competes for receptor sites with natural histamine, a biogenic amine present in most body cells and tissues[5].

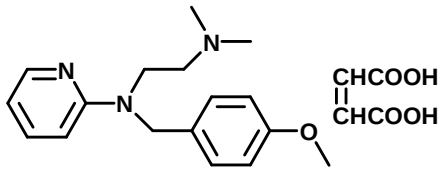

Figure 2: Pyrilamine {1, 2-Ethane diamine *N*-[(4-methoxy phenyl) methyl]-*N*^{1}, *N*^{1}-dimethyl-*N*-2piridinyl- (Z)-2 butene dioate (1:1) (or) 2-[(2-Dimethyl amino) ethyl) (*p*-methoxy benzyl) amino] pyridine maleate (1:1) [59-33-6]}

A very few Physico-chemical methods appeared in the literature for the assay of BUCZ and PYRA in biological fluids, and pharmaceutical formulations. Most

of them are based on HPLC[6-10,18], TLC[11], GLC[12-15] and visible spectrophotometric methods[5,16,18-20]. The analytically useful functional groups in BUCZ and PYRA have not been fully exploited for designing suitable visible spectrophotometric methods and so still offer a scope to develop few more visible spectrophotometric methods with better sensitivity, selectivity, precision and accuracy.

The author has developed a simple and sensitive UV spectrophotometric method in $CHCl_3$ for the estimation of BUCZ and PYRA in pure or pharmaceutical formulations and adopted it as a reference method to compare the results obtained with the proposed methods.

EXPERIMENTAL

An Elico UV–Visible digital spectrophotometer with 1cm matched quartz cells were used for the spectral and absorbance measurements, an Elico LI-120 digital pH meter was used for pH measurements. All the chemicals and reagents used were of analytical grade and the aqueous solutions were freshly prepared with triple distilled water.

Preparation of the reagents

Tpooo (0.2%) (5.70×10^{-3}M) was prepared by dissolving 200 mg of Tropaeolin ooo in 100 ml of distilled water[1]. HCl solution was prepared by dissolving 8.6 ml of conc. HCl to 1000 ml of distilled water and Standardized.

Preparation of standard solutions

A 1 mg/ml individual solutions were prepared by dissolving 100 mg of pure BUCZ in 5 ml of 0.1N HCl, and dissolving 100 mg of pure PYRA in 10ml of distilled water followed by dilution to 100 ml with distilled water and the stock solution was diluted step wise with distilled water to get the working standard solutions of concentrations of 25 μg/ml.

Method

Into a series of 125 ml separating funnels containing aliquots of standard BUCZ and PYRA solution (0.5-3.0 ml, 25 μg/ml), 6.0 ml of 0.1 M HCl solution and 2.0 ml of dye (Tpooo) solution were added successively. The total volume of aqueous phase in each separating funnel was adjusted to 15.0 ml with distilled water. To each separating funnel 10.0 ml of chloroform was added and the contents were shaken for 2 min. The two phases were allowed to separate and the absorbance of the separated chloroform layer was measured at 480 nm against a similar reagent blank. The amount of BUCZ and PYRA were deduced from the calibration graphs.

For pharmaceutical formulations

An accurately weighed portion of tablet powder equivalent to about 100 mg of BUCZ and PYRA were transferred into a 100 ml volumetric flask. Added about 80 ml of warm chloroform and shaken well for about 20 minutes. The contents were diluted with chloroform upto the mark and mixed thoroughly. The solution was filtered the filtrate was evaporated to dryness. The residue was used for the preparation of standard solution as shown under standard solution preparation. These solutions were analyzed as under procedures described from bulk solutions.

RESULTS AND DISCUSSION

As BUCZ and PYRA possess two tertiary nitrogens, it forms an ion association complex with an acid dye (Tpooo) which is extractable in to chloroform from aqueous phase. Each protonated nitrogen (positive charge) of BUCZ and PYRA as hydrochloride is expected to attract the oppositely charged part (negative charge) of dye and be have as a single unit being held together by electrostatic attraction. It is

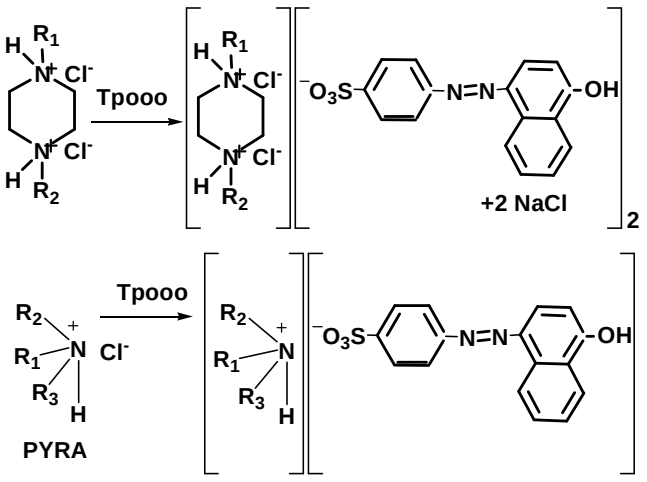

Figure 3

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supported by slope-ratio method. Based on the analogy the structures of ion association complexes are shown in Figure 3

The optical characteristics such as Beer's law limits, absorption maxima, Sandell's sensitivity, molar extinction coefficient, percent relative standard deviation and percent range of error (0.05 level and 0.01 confidence limits) were calculated for the method and the results are summarized in TABLE 1. The regression analysis using the method of least squares was made for the slope (b), intercept (a) and correlation (R) obtained from different concentrations and the results are summarized in TABLE 1. The optimum conditions for the colour development were established by varying the parameters one at a time in each method, keeping the others fixed and observing the effect produced on the absorbance of the coloured species. The values obtained for the determination of BUCZ, PYRA in tablets by the proposed and UV method is compared in TABLE 2. To evaluate the validity and reproducibility of the method, known amounts of pure drug were added to previously analyze pharmaceutical preparations and the mixtures were analyzed by the proposed methods. The percent recoveries are given in TABLE 2.

TABLE 1 : Optical and regression characteristics, precision and accuracy of the proposed method for two drugs

Parameter	BUCZ	PYRA
λ_{max} (nm)	480	480
Beer's law limits (μg/ml)	1.25-7.5	1.25-7.5
Detection limit (μg/ml)	4.034	1.881
Molar absorptivity ($1\ mol^{-1}.cm^{-1}$)	1.609×10^4	2.531×10^4
Sandell's sensitivity ($\mu g.cm^{-2}$ /0.001 absorbance unit)	9.961×10^{-2}	6.184×10^{-2}
Optimum photometric range (μg/ml)	3.0-7.5	2-6.3
Regression equation (Y=a+bc) slope (b)	0.03437	0.06723
Standard deviation on slope (S_b)	1.026×10^{-2}	9.678×10^{-3}
Intercept (a)	2.75×10^{-3}	3.25×10^{-3}
Standard deviation on intercept (S_a)	4.52×10^{-2}	4.011×10^{-2}
Standard error on estimation (S_e)	4.055×10^{-2}	3.825×10^{-2}
Correlation coefficient (r)	0.9998	0.9999
Relative standard deviation (%)	0.1362	0.9363
% Range of error (confidence limits)		
0.05 level	0.1566	1.076
0.01 level	0.2455	1.688

*** Average of six determinations considered**

TABLE 2 : Assay of two drugs in pharmaceutical formulations

Formulations*	Amount taken (mg)	Amount found by proposed Method**		Reference method		% recovery by proposed method***	
	BUCZ & PYRA	BUCZ	PYRA	BUCZ	PYRA	BUCZ	PYRA
Tablet I	25	24.68 ± 0.51 F = 2.048 t = 0.7821	24.93 ± 0.25 F = 2.822 t = 1.344	24.96 ± 0.73	25.19 ± 0.42	99.73 ± 0.61	99.91 ± 0.72
Tablet II	25	24.63 ± 0.43 F=2.285 t=0.8980	24.78 ± 0.45 F = 1.837 t = 0.6209	24.91 ± 0.65	24.97 ± 0.61	99.83 ± 0.33	99.88 ± 0.44
Tablet III	25	24.59 ± 0.32 F = 2.540 t = 1.210	24.59 ± 0.34 F = 2.712 t =1.270	24.88 ± 0.51	24.92 ± 0.56	99.65 ± 0.91	99.56 ± 0.84
Tablet IV	25	24.72 ± 0.31 F = 2.2018 t = 1.124	24.73 ± 0.53 F = 1.694 t = 0.482	24.97 ± 0.46	24.90 ± 0.69	99.83 ± 0.90	99.54 ± 0.32

*** Tablets from four different pharmaceutical companies**

**** Average ± standard deviation of six determinations, the t-and F-test values refer to comparison of the proposed method with the reference method. Theoretical values at 95% confidence limit, F = 5.05, t = 2.57**

***** Recovery of 10mg added to the pre-analyzed pharmaceutical formulations (average of three determinations)**

CONCLUSIONS

The developed UV Spectrophotometric method for the estimation of BUCZ and PYRA was found to be simple and useful with high accuracy, precision, and reproducible. Sample recovery in all formulations using the above method was in good agreement with their respective label claim or theoretical drug content, this suggesting the validity of the method and non interference of formulation excipients in the estimation.

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