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Simple spectrophotometric determination of desloratadiene from pharmaceutical formulation

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ABSTRACT

Simple sensitive and accurate extractive spectrophotometric methods have developed for the estimation of desloratadine in pharmaceutical dosage form. The methods are based on the formation of coloured complexes by the drug with reagents like bromophenol blue, solochrome dark blue, bromocresol green and congo red in acidic medium. The ion associated complexes were formed and quantitatively extracted under the experimental condition in chloroform. The absorbance values were measured at 420 nm, 495 nm, 430 nm and 400 nm respectively. The proposed methods were validated statistically. Recoveries of methods were carried out by standard addition methods. The linearity was found to be 1.0-5.0μg/ml, 2.5-25μg/ml, 2.5-30μg/ml and 10-60μg/ml for methods 1, 2, 3 and 4 respectively. The low values of standard deviation and percentage RSD indicate high precision of methods. Hence these methods are useful for routine estimation of desloratadine in tablets.

KEYWORDS

Desloratadine;
Bromophenol blue;
Solochrome dark blue;
Bromocresol
Green;
Congo red.

INTRODUCTION

Desloratadine is 8-chloro-6,1 dihydro-11(4 piperi dinylidine)-5-H-benzo[1,2-b]pyridine, descarbo ethoxylatadine. It shows molecular formula as $C_{19}H_{19}ClN_2$ with molecular weight 310.82. It is not yet official in any pharmacopeia. It is non sedating peripheral histamine H_1 receptor antagonist, active metabolite of loratadine. A literature survey reveals spectrophotometric[1-3] and HPLC[4-8] methods. The proposed methods involve formation of ion pair complexes of desloratadine with bromophenol blue, solochrome dark blue, bromocresol green and congo red in acidic medium.

MATERIALS AND METHODS

A SHIMADZU -160 A double beam UV-VISIBLE recording spectrophotometer with pair of 10 mm matched quartz cell was used to measure absorbance of solutions. A SHIMADZU analytical balance was used.

Bromophenol blue, solochrome dark blue, bromocresol green, congo red, hydrochloric acid, potassium hydrogen phthalate and chloroform of A.R. grade were used in the study.

Prepration of standard solution and reagents

Stock solution of desloratadine (100 μg/ml) was prepared in ethanol. From this stock solution working standard (10 μg/ml) was prepared by diluting 10 ml

stock solution to 100 ml with ethanol. 0.1% w/v solution of bromophenol blue, 0.25% w/v solochrome dark blue 0.02% w/v bromocresol green and 0.6 % w/v congo red solutions were prepared in distilled water respectively.

Potassium hydrogen phthalate buffer solution of pH 4.01 was prepared in distilled water. Dilute hydrochloric acid was used to adjust desired pH of buffer solution.

EXPERIMENTAL

Method 1(with bromophenol blue)

Into a series of separating funnels appropriate amount of the working standard drug solutions were pipetted out. To each funnel 1.0 ml of buffer (pH = 4.2) and 6.0 ml of 0.1% w/v bromophenol blue were added. 10 ml of chloroform was added to each funnel. The solutions were shaken for thorough mixing of the two phases and were allowed to stand for clear separation of the layers.

The absorbance values of the chloroform layers were measured against their respective reagent blank at the wavelength of the maximum absorbance (λ_{max} 420 nm).

Method 2(with solochrome dark blue)

Into a series of separating funnels appropriate amount of the working standard drug solutions were pipetted out. To each funnel 4.0 ml of buffer (pH = 1.10) and 4 ml of 0.25% w/v solochrome dark blue were added. 10 ml of chloroform was added to each funnel. The solutions were shaken for thorough mixing of the two phases and were allowed to stand for clear separation of the layers.

The absorbance values of the chloroform layers were measured against their respective reagent blank at the wavelength of the maximum absorbance (λ_{max} 495 nm).

Method 3(with bromocresol green)

Into a series of separating funnels appropriate amount of the working standard drug solutions were pipetted out. To each funnel 1.0 ml of buffer (pH = 3.7) and 5.5 ml of 0.02% w/v bromocresol green were added. 10 ml of chloroform was added to each funnel. The solutions were shaken for thorough mixing of the two phases and were allowed to stand for clear separation of the layers.

The absorbance values of the chloroform layers were measured against their respective reagent blank at the wavelength of the maximum absorbance (λ_{max} 430 nm).

Method 4 (with congo red)

Into a series of separating funnels appropriate amount of the working standard drug solutions were pipetted out. To each funnel 1.0 ml of buffer (pH = 3.7) and 5.0 ml of 0.06% w/v congo red were added. 10 ml of chloroform was added to each funnel. The solutions were shaken for thorough mixing of the two phases and were allowed to stand for clear separation of the layers.

The absorbance values of the chloroform layers were measured against their respective reagent blank at the wavelength of the maximum absorbance (λ_{max} 400 nm.)

Estimation from tablets

Twenty tablets of labelled claim 5 mg of desloratadine were weighed accurately. Average weight of each tablet was determined. Tablets were crushed into fine powder. An accurately weighed quantity of powder equivalent to 10 mg of desloratadine was transferred into a beaker and it was shaken with 50 ml of ethanol and filtered. The filtrate and the washing were collected in a 100.0 ml volumetric flask. This filtrate and the washing were diluted up to the mark with ethanol to obtain final concentration as 100 μg/ml. This solution was further diluted to give 10 μg/ml. Such solution was used for methods (1-4) respectively.

Appropriate aliquots of drug solution were taken and the individual assay procedures were followed for the estimation of drug contents in tablets. The concentration of the drug in the tablets was calculated using calibration curve. The recovery experiment was carried out by standard addition method. Results of analysis are given in TABLE 1.

RESULTS AND DISCUSSION

The extractive spectrophotometric methods are popular due to their sensitivity in assay of the drug and

hence ion pair extractive spectrophotometric methods have gain considerable attention for quantitative determination of many pharmaceutical preparations. These proposed methods are extractive spectrophotometric methods for the determination of desloratadine by using chloroform as solvent from its formulations i.e. tablets.

The colour ion pair complexes formed are very stable. The working conditions of these methods were established by varying one parameter at time and keeping the other parameters fixed by observing the effect produced on the absorbance of the colour species. The various parameters involved for maximum colour development for these methods were optimized. The proposed methods were 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 % RSD (relative standard deviation). The optical characteristics such as absorption maxima (nm), molar absorptivity (l - mole^{-1} cm^{-1}), co-rrelation coefficient (r) and sandell sensitivity ($\mu g/cm^2/0.001$) were calculated and are also summarized in TABLE 1. Assay results of recovery studies are given in TABLE 2.

Results are in good in agreement with labelled value. The percent recovery obtained indicates non interference from the common excipients used in the formulation. The reproducibility, repeatability and accuracy of these methods were found to be good, which is evidenced by low standard deviation.

The proposed methods are simple, sensitive, accurate, precise and reproducible. They are directly applied to drug to form chromogen. Hence they can be successfully applied for the routine estimation of desloratadine in bulk and pharmaceutical dosage form even at very low concentration and determination of stability of drug in formulation such as tablets.

The strong recommendation is made here for the proposed methods for determination of desloratadine from its formulation.

TABLE 1 : Optical and regression of drug in different methods

Parameter	Methods			
	1	2	3	4
λ max (nm)	420	495	430	400
Beer law limits (μg/mL)	1.0-5.0	2.5-25	2.5-30	10-60
Molar absorptivity (l/mol.cm)	4.494×10^4	8.889×10^3	9.821×10^3	5.066×10^3
Sandell's sensitivity	0.144	2.88 $y10^{-2}$	$3.16x10^{-2}$	1.62×10^{-2}
Correlation coefficient(r^2)	0.9999	0.9984	0.9999	0.9999
Regression equation (y=b+ac)				
Slope (a)	0.1446	0.0286	0.0316	0.0163
Intercept	-0.0013	0.0005	-0.0005	0.0005

TABLE 2: Results of recovery of drug

Reagent	Amount of drugadded μg/ ml	Amount of standard drugμg/ml	Total amount recovered	Percentage recovery	Standard deviation	Percentage of relative standard deviation
Bromo phenol blue	1.0	0	0.9978	99.780	0.00501	0.5025
	1.0	0.5	1.4979	99.860	0.00526	0.3513
	1.0	1.0	1.9968	99.840	0.006593	0.3302
	1.0	1.5	2.5025	100.10	0.006373	0.2546
Solochrome dark blue	2.5	0.0	2.494	99.760	0.03706	1.4856
	2.5	2.5	4.990	99.800	0.02570	0.5150
	2.5	5.0	7.489	99.853	0.03967	0.5297
	2.5	7.5	9.9935	99.935	0.03567	0.3569
Bromocresol green	2.5	0	2.494	99.760	0.003539	1.418
	2.5	2.5	4.987	99.740	0.00275	0.5517
	2.5	5.0	7.501	100.01	0.003579	0.4771
	2.5	7.5	9.997	99.97	0.00287	0.2871
Congo red	5.0	0.0	4.991	99.827	0.0660	1.3223
	5.0	5.0	9.991	99.91	0.0425	0.4254
	5.0	10.0	14.973	99.82	0.0601	0.4013
	5.0	15.0	19.982	99.91	0.04672	0.2338

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