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SIMPLE EXTRACTIVE SPECTROPHOTOMETRIC DETERMINATION OF DESLORATADINE FROM PHARMACEUTICAL FORMULATION

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ABSTRACT

Simple sensitive and accurate extractive spectrophotometric methods was developed for the estimation of desloratadine in pharmaceutical dosage form. The methods were based on the formation of coloured complexes by the drug with reagents like eriochrome black T, methyl orange and picric acid in acidic medium. The ion associated complexes was formed. They were quantitatively extracted under the experimental condition in chloroform. The absorbance values were measured at 500 nm, 420 nm and 414 nm, respectively. The proposed methods were validated statistically. Recoveries of methods were carried out by standard addition methods. The Beer's law range was found to be 5-50 µg/mL, 1-5 µg/mL and 2.5-30 µg/mL for methods I, II and III, 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.

Key words : Desloratadine, Eriochrome black T, Picric acid, Methyl orange, Spectrophotmetric, Extractive.

INTRODUCTION

Desloratadine is 8-chloro-6, 1 dihydro-11(4 piperidinylidine) -5-H- benzo [1, 2-b]pyridine, descarboethoxylatadine. It shows molecular formula as $C_{19}H_{19}ClN_2$ with molecular weight 310.82. It is not yet official in any pharmacopeias. 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 eriochrome black T, methyl orange and picric acid in acidic medium.

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EXPERIMENTAL

Material 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. Eriochrome black T, picric acid, methyl orange, potassium hydrogen phthalate and chloroform of A. R. grade were used in the study.

Prepration of standard solution

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

Prepartion of reagents

0.1% w/v solution of eriochrome black T, 0.05 % w/v picric acid and methyl orange solutions were prepared by dissolving the reagent in 2 mL of ethanol and volume was made up by using distilled water, respectively.

Prepartion of buffer solution

Potassium hydrogen phthalate buffer solution of pH 4 was prepared in distilled water and different acidic buffer solutions were prepared by addition of hydrochloric acid.

EXPERIMENTAL

Method I: Into a series of separating funnels, appropriate amount of the working standard drug solutions were pipetted out. To each funnel 1 mL of buffer solution of pH as 3.2 and 6.0 mL of 0.1 % w/v eriochrome black T solution was 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 separation of the layers.

The absorbance value of the chloroform layer was measured against their respective reagent blank at the wavelength of the maximum absorbance (λ_{max} 500 nm).

Method II: 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 solution of pH

3.3 and 3 mL of 0.05 % w/v methyl orange solution 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 separation of the layers. The absorbance value of the chloroform layer was measured against their respective reagent blank at the wavelength of the maximum absorbance (λ_{max} 420 nm).

Method III : Into a series of separating funnels, appropriate amount of the working standard drug solutions were pipetted out. To each funnel 1 mL of buffer solution of pH 3.5 and 1 mL of 0.05 % w/v picric acid solution 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 separation of the layers. The absorbance value of the chloroform layer was measured against their respective reagent blank at the wavelength of the maximum absorbance (λ_{max} 414 nm.)

Estimation from tablets

Twenty tablets of labelled claim 5 mg of desloratadine were weighed accurately. Average weight of each tablet was determined. Twenty 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. Ten mL of this solution was further diluted to give 10 µg /mL. This 10 µg/mL of solution was further used for methods I, II and III, respectively.

Table 1 : Optical and regression of drug in different methods

Parameter	Method		
	I	II	III
λ max (nm)	500	420	414
Beer law limits (mcg/mL)	5-50	1-5	2.5-30
Molar absorptivity(L/mol. cm)	5.9988 x 10^3	2.828 x10^4	7.3975 x 10^3
Sandell's sensitivity	1.92 x10^{-2}	9.2 x10^{-2}	2.48 x10^{-2}
Correlation coefficient (r^2)	0.9999	0.9997	0.9999

Cont…

Parameter	Method		
	I	II	III
Regression equation (y = b + ac)			
Slope (a)	0.0193	0.091	0.0248
Intercept	-0.0005	0.0005	0.0005

Appropriate aliquots of drug solution were taken. The individual assay procedures was carried out 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. The values of optical and regression terms of analysis are given in Table 1.

Table 2: Results of recovery of drug (Method I)

Amt. of drug added (µg/mL)	Amt. of stand. added (µg/mL)	Total amount recovered	Recovery (%)	Stand. deviation	% of relative stand. Deviation C. O. V.	Mean stand. deviation	Mean of C. O. V.
5	0	4.9925	99.851	0.0701	1.4033		
5	5	9.9258	99.925	0.0697	0.6975	0.0698	0.7291
5	10	14.9925	99.950	0.0700	0.4671		
5	15	19.9925	99.962	0.0697	0.3487		
Mean of percent (%) recovery = 99.922							

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 gained 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 viz. tablets.

The colour ion–pair complexes are formed and 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.

Table 3 : Results of recovery of drug (Method II)

Amt. of drug added (µg/mL)	Amt. of stand. added (µg/mL)	Total amount recovered	Recovery (%)	Stand. deviation	% of relative stand. deviation C. O. V.	Mean stand. deviation	Mean of C. O. V.
5	0	4.9952	99.525	0.01886	1.8953		
5	1	1.9999	99.950	0.01410	0.70568	0.01498	0.8493
5	2	2.9984	99.946	0.01473	0.49147		
5	3	3.9968	99.920	0.01223	0.30604		
Mean of percent (%) recovery = 99.835							

Table 4 : Results of recovery of drug (Method III)

Amt. of drug added (µg/mL)	Amt. of stand. added (µg/mL)	Total amount recovered	Recovery (%)	Stand. deviation	% of relative stand. deviation C. O. V.	Mean stand. deviation	Mean of C. O. V.
2.5	0	2.4884	99.5393	0.04484	1.80217		
2.5	2.5	4.9942	99.8851	0.05380	0.07738	0.05430	1.0703
2.5	5.0	7.4942	99.9235	0.06431	0.8588		
2.5	7.5	9.9942	99.9422	0.05423	0.5427		
Mean of percent (%) recovery = 99.8225							

The proposed methods were validated statistically and by recovery studies. The molar absorptivity and Sandell's sensitivity values (Table 1) 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}), correlation coefficient (r) and Sandell sensitivity (µg/cm^2/0.001) were calculated and are also summarized in Table 1. Assay results of recovery studies are given in Tables 2, 3 and 4. 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 values of standard deviations. Methods suggested in literature (applied in UV region), need costly reagent for development of chromogen and useful in higher concentrations. The only proposed methods are simple, sensitive, accurate, precise and reproducible applicable to even very low concentrations as compared to previous methods suggested in literature. 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 in formulation such as tablets.

The proposed methods are strongly recommended for determination of desloratadine from its formulation viz. tablets.

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REFERENCES

1. J. M. Patel, G. S. Talele, R. A. Fursule, Asian J. Chem., **16(2)**, 1220-1222, (2004).

2. J. M. Patel, G. S. Talele, R. A. Furule and S. J. Surana, Indian Drugs, **43(6)**, 507-508 (2006).

3. S. Caglar and A. Oztune, J. AOAC, Int., **90 (2)**, 372-375 (2007).

4. Nahed El-Enany, Dina El-Sherbiny and Fathalla Belel., Chem. Pharm. Bull., **55(12)**, 1662-1670 (2007).

5. L. H. Liu, M. L. Qi, P. Wang and H. Z. Li, J. Pharm. Biomed. Anal., **34(5)**, 1013-1019 (2004).

6. A. R. More, A. J. Vaidya, V. V. Vaidya and R. G. Deshmukh, Indian Drugs, **42(8)**, 525-529 (2005).

7. Dina T. El-Sherbiny, Nahed El-Enany, Fathalla F. Belal and Steen H. Hansen, J. Pharm. and Biomed., Anal., **43(4)**, 1236-1242 (2007).

8. F. C. W. Sutherland, A. D. De Janger and A. F. Hundt, J. Chromatogr. A, **914 (12)**, 37-43 (2001).

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