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Simultaneous determination of loratadine and pseudoephedrine sulfate in pharmaceutical formulation by RP-LC and derivative spectrophotometry☆

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Abstract

Highly sensitive, simple and accurate reversed phase liquid chromatographic and first derivative spectrophotometric methods for determination of antihistaminic drug loratadine [I] and nasal decongestant drug pseudoephedrine sulfate [II] are described. The HPLC method involves separation of [I] and [II] on μ-BondaPak C18 column using mixture of (methanol:H_2O:phosphoric acid:ammonium dihydrogen phosphate) (220:300:2:3 g) (V/V/V/W), 60 and 40% acetonitrile as mobile phase flowing at 2 ml/min with ultraviolet detection at 247 nm. The calibration graphs are linear from 5 to 100 μg/ml for [I] and from 120 to 1200 μg/ml for [II] the detection limits are 0.5 μg/ml for [I] and 60 μg/ml for [II]. The spectrophotometric method is based on recording the first derivative spectra for [I] and [II] at 307, 266 nm, respectively, of their solutions in 0.1 M hydrochloric acid using the acid as blank. The calibration graphs are linear in the range of 5–25 μg/ml for [I] and 240–720 μg/ml for [II]; the limits of detection are 0.16 μg/ml for [I] and 10 μg/ml for [II]. The mean percentage recoveries obtained for different synthetic mixtures by using this method are 97.6% with coefficient of variation 1.79 for [I] and 101.6% with coefficient of variation 1.95 for [II]. The two methods have been applied successfully for the determination of [I] in its combination with [II] Clarinase® tablets and [I] alone in different pharmaceutical dosage forms.

Keywords: Loratadine; Pseudoephedrine sulfate; Derivative spectrophotometry; Reversed Phase liquid chromatography; Dosage forms

1. Introduction

Direct UV-absorbance measurement is subjected to interference from co-formulated drugs, excipients and/or degradation products. Derivative spectrophotometry is analytical technique of great

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utility for extracting both qualitative and quantitative information from spectra composed of unresolved bands. It tends to emphasize subtle spectral features by representing them in a new and visually more accessible way allowing the resolution of multicomponent systems and minimizing the effect of spectral background interferences in pharmaceutical application [1–7]. Derivative spectrophotometry has led to significant developments in the analysis of drugs in the presence of their degradation products or in multicomponent mixtures [8].

Loratadine [I] and pseudoephedrine sulfate [II] are present together in dosage form prescribed to relieve symptoms of allergic rhinitis. Different analytical procedures have been reported for the determination of each one of them alone. No methods have been reported for their simultaneous quantification in their mixtures.

Loratadine is a long acting non-sedating antihistamine. Different methods were developed for determination of [I] including polarographic methods [9,10], spectrophotometric methods [11–14]. Different chromatographic methods have been developed for the determination of [I] and its metabolite in human plasma. These include GC [15,16], HPLC [17,18].

A densitometric method was developed for determination of [I] in pharmaceutical preparations [19]. Pseudoephedrine is a sympathomimetic drug, which occurs naturally in plants of genus Ephedra. Different analytical procedures for its determination of [II] have been reviewed in the analytical profile of drug substances [20].

Several other methods have been reported include: determination of pseudoephedrine HCl in tablets and liquid formulation by biphasic titration [21]. USP 24 describes a non-aqueous titration method for determination of pseudoephedrine sulfate.

Several spectrophotometric methods have been developed for determination of pseudoephedrine hydrochloride either alone [22,23] or in combined preparation such as with chlorpheniramine maleate using first derivative spectrophotometry on a diode array spectrometer [24] or with chlorpheniramine and dextromethorphan using second derivative photodiode array spectroscopy [25], or with triprolidine and dextromethorphan using derivative difference spectrometry [26], or with acrivastine using second derivative and ratio spectra first derivative methods [27] or with dextromethorphan hydrobromide using second derivative spectrophotometry and multi wavelength spectrophotometry [28]and recently pseudoephedrine sulfate was determined with loratadine and dexobromopheniramine using ratio spectra derivative spectrophotometry [29].

Several chromatographic methods have been applied for the determination of [II] including: TLC [30], GC [31–34], HPLC [35–39], HPTLC [40].

Recently,Chen described a capillary zone electrophoretic method for determination of pseudoephedrine [41]. A proton nuclear magnetic resonance spectroscopic method has been developed for determination of [II] in dosage forms [42]. A stereospecific radioimmunoassay was used for determination of [II] in human plasma [43].

The commercially available dosage form Clarinase® contains[I] and [II] with the wide difference in concentrations (1:24) and no method has been reported for their simultaneous determination. Thus the aim of this study is to establish a simple method for their simultaneous determination suitable for quality control purposes.

2. Experimental

2.1. Materials

Authentic samples of loratadine and pseudoephedrine sulfate were kindly supplied by Sigma Company for Pharmaceutical Industries, Quesna, Menofia, Egypt.

All pharmaceutical preparations were purchased from local market. They include: Clarinase® tablets claimed to contain 5 mg loratadine and 120 mg pseudoephedrine sulfate. Mosedin®, Claritine® and Loratan® tablets; they claimed to contain 10.0 mg loratadine.

Loratan® and Mosedin® syrups; both claimed to contain 1 mg/ml loratadine.

The internal standard references; Methylparaben (MPB) (Sigma). Propylparaben (PPB) (Sigma).

Hydrochloric acid analytical grade, methanol and acetonitrile HPLC grade (BDH). Phosphoric acid (Merck). Ammonium dihydrogen phosphate (Riedel).

2.2. Spectrophotometric equipment and conditions

A shimadzu UV-160 double beam spectrophotometer. The derivative UV spectra were recorded in 1 cm quartz cells over the range 220–400 nm.

2.3. Chromatographic system and conditions

HPLC measurements were performed on Waters Instrument equipped with a model 600 Pump U6K-injector and 486 UV detector.

Separation was performed on μ-BondaPak C18 (300 × 3.9 mm, 10 μm) column. The mobile phase consisted of mixture of H_2O:methanol:phosphoric acid:Ammonium dihydrogen phosphate (300:220:2:3 g) (v/v/v/w), 60 and 40% acetonitrile.

The mobile phase was prepared daily, filtered, sonicated before use and delivered at a flow rate 2 ml/min. The detector wavelength was set at 247 nm.

2.4. Preparation of calibration curves

2.4.1. Derivative spectrophotometric method

2.4.1.1. Loratadine. A stock solution was prepared in 0.1 M HCl with a concentration 0.5 mg/ml. Different aliquots of loratadine stock solution were taken and diluted with 0.1 M HCl to produce concentrations ranging from 5 to 25 μg/ml.

2.4.1.2. Pseudoephedrine sulfate. A stock solution of pseudoephedrine sulfate with a concentration 2.4 mg/ml was prepared in 0.1 M HCl.

Different aliquots of this solution were taken and diluted with 0.1 M HCl to produce concentrations ranging from 240 to 720 μg/ml. The first order derivative spectra were recorded using the prepared solutions against 0.1 M HCl as blank.

The zero-crossing points of derivative spectra of loratadine and pseudoephedrine sulfate were assigned.

The values of the first derivative absorbance ^1D for either compound were obtained using five different concentrations by measuring each concentration against solvent blank at the chosen wavelength; 307 and 266 nm for loratadine and pseudoephedrine, respectively.

The values of ^1D were plotted against corresponding concentrations.

2.4.2. HPLC method

2.4.2.1. Loratadine. A stock solution of loratadine was prepared in the mobile phase to contain 1 mg/ml. Aliquots of this solution were diluted with the mobile phase to obtain solutions having concentrations from 5 to 100μg/ml each contains 5 μg/ml MPB as internal standard.

2.4.2.2. Pseudoephedrine sulfate. A stock solution of pseudoephedrine sulfate 2.4 mg/ml was prepared in the mobile phase. Aliquots of this solution were diluted with the mobile phase to obtain solutions having concentrations from 120 to 1200 μg/ml each contains 5 μg/ml MPB.

Separately inject equal volumes (about 20 μl) of different solutions into the chromatograph, record the chromatograms and calculate the ratio of the response of each solution to that of internal standard and construct the calibration curves.

To study the accuracy and precision of the proposed methods, recovery experiments were carried out on different synthetic mixtures of loratadine and pseudoephedrine at different ratios including the ratio (1:24) as present in Clarinase® tablets.

The proposed methods were applied for the analysis of synthetic mixture of each drug in presence of common tablet excipients as magnesium stearate, talc, starch and lactose to assure the specificity of the method.

2.5. Procedures for pharmaceutical dosage forms

2.5.1. Derivative spectrophotometric method

2.5.1.1. For analysis of Clarinase® tablets. Ten tablets were finely powdered, weighed, a portion equivalent to 10 mg of loratadine was suspended in 0.1 M HCl, transferred quantitatively into 50 ml volumetric flask, sonicated for 15 min, complete to volume with 0.1 M HCl and filtered. Dilute 5 ml of filtrate to 50 ml with 0.1 M HCl. Record the first derivative spectrum of this solution, determine ^{1}D at 307 and 266 nm, and get the contents of loratadine and pseudoephedrine sulfate using the linear regression equations of calibration curves.

2.5.1.2. For analysis of Mosedin®, Loratan® and Claritine® tablets. Ten tablets were finely powdered, weighed, a portion equivalent to 20 mg of loratadine was suspended in 0.1 M HCl, transferred quantitatively into 50 ml volumetric flask, sonicate complete to volume with 0.1 M HCl filter, dilute 5 ml of filtrate to 100 ml with 0.1 M HCl. Determine ^{1}D at 307 nm. The concentration of loratadine in tablets was determined by reference to the standard calibration curve or a standard sample was analyzed simultaneously.

2.5.2. HPLC method

2.5.2.1. For Clarinase®, Mosedin®, Loratan® and Claritine® tablets. Ten tablets were finely powdered, weighed, a portion equivalent to 10 mg of loratadine was suspended in the mobile phase, transferred quantitatively into 50 ml volumetric flask, sonicate for 15 min, complete to volume with the mobile phase filter and then dilute 1.0 ml of filtrate to 10.0 ml after adding 1.0 ml of MPB (50 μg/ml) as internal standard.

2.5.2.2. For Loratan® and Mosedin® syrups. Dilute 1.0 ml of the syrup to 10.0 ml with the mobile phase, place 2.0 ml of this solution and 1.0 ml PPB (100 μg/ml) into 10.0 ml volumetric flask and complete to volume with the mobile phase. An aliquot (20 μl) was chromatographed by HPLC.

The concentration of loratadine and pseudoephedrine sulfate in Clarinase® and loratadine concentrations in other dosage forms were calculated from the linear regression equations of calibration curves or using reference standard solution injected under the same conditions.

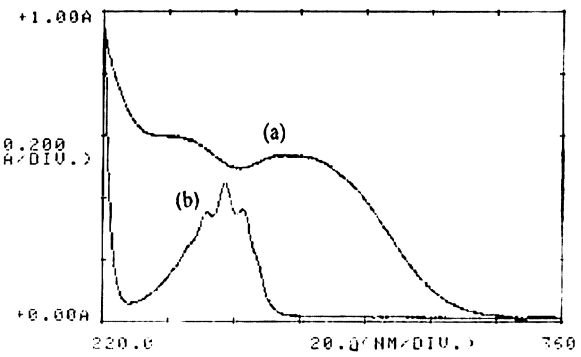

Fig. 1. Zero-order UV spectra of 20 μg/ml Loratadine (a), 480 μg/ml pseudoephedrine sulfate (b) in 0.1 M HCl.

2.6. Procedures for preparation of degradation product of loratadine

Reflux 10 mg of pure drug with 10 ml 6 N HCl in boiling water bath for 2.5 h. Adjust the degradate solution to pH 8–9 using 6 N NaOH and transfer quantitatively into a separating funnel. Extract three times by shaking each time with 15 ml diethyl ether. Collect the etherial extract and evaporate to dryness at 40 °C. Dry the residue in vacuum dessicator for 30 min. Dissolve the residue in 50 ml 0.1 N HCl. Inject 20 μl of this solution into the chromatograph.

3. Results and discussion

Fig. 1 shows the zero-order UV spectra of loratadine [I] and pseudoephedrine sulfate [II]. It is quite clear that there is extensive spectral overlap thus, conventional UV spectrophotometry can not be used for the individual determination of either drug in their binary mixture. The study of their derivative spectra may overcome this difficulty; thus their first derivative spectra were obtained.

The first derivative spectrum of loratadine exhibits a maximum at 307 nm while the pseudoe-

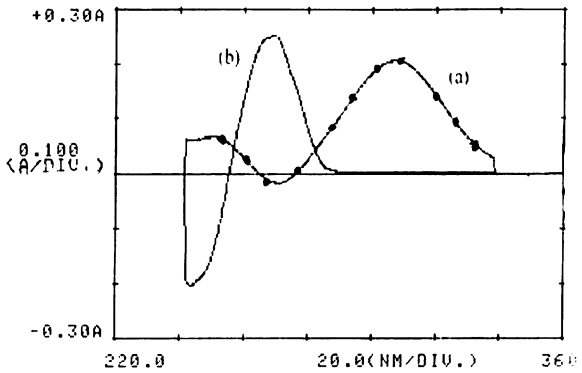

Fig. 2. First- order derivative spectra of 20 μg/ml Loratadine (a), 480 μg/ml pseudoephedrine sulfate (b) in 0.1 M HCl.

phedrine reads zero and pseudoephedrine sulfate exhibits an absorption at 266 nm while loratadine reads zero, Fig. 2. Quantitative investigations using regression analysis have established that the concentration of loratadine and pseudoephedrine sulfate correlates very well with the measured first derivative peaks as shown in Table 1.

The recovery test was performed using synthetic mixtures containing various amounts of [I] and [II]. The results are reproducible and precise as C.V. are very low (Table 2).

This method has been successfully applied to Loratan®, Claritine®, Mosedin® and Clarinase® tablets and the results obtained are shown in Tables 5 and 6. In case of tablets the excipients show no interference.

For HPLC method, initially various mobile phase compositions were tried in attempts to separate the drugs and internal standard HPLC system using C18 column and 60% (water:methanol:phosphoric acid:ammonium dihydrogen phosphate) (300:220:2:3 g) and 40% acetonitrile gave good separation.

Fig. 3A, B show typical HPLC chromatograms of standard mixture of the two drugs and their

Table 1
Quantitative parameters for the determination of loratadine and pseudoephedrine sulfate with first derivative spectrophotometric method

Compound	Linearity range (μg/ml)	Intercept	S.E.M.	Slope	S.E.M.	Correlation coefficient	LOD (μg/ml)
Loratadine	5–25	0.0033	0.00014	0.0102	0.00006	0.9997	0.16
Pseudoephedrine sulfate	240–720	0.0049	0.00068	0.0005	0.00016	0.9993	10.0

Table 2
Recovery of loratadine and pseudoephedrine sulfate in synthetic mixtures by the proposed first derivative spectrophotometric method[a]

Loratadine			Pseudoephedrine sulfate		
Added (μg/ml)	Found (μg/ml)	% Recovery	Added (μg/ml)	Found (μg/ml)	% Recovery
13	14.7	97.7	240	231.9	96.6
15	14.5	96	360	368.2	102.3
15	14.7	97.7	480	482.1	100.4
15	14.7	97.7	600	618	103.1
5	4.9	97.7	240	248	103.3
10	9.4	94.4	240	242	100.8
15	14.5	96.5	240	250	104.2
20	19.4	97.0	240	244	101.7
25	25.1	100.5	240	244	101.7
20	20.1	100.4	480	490.3	102.1
Mean % recovery ± S.D., C.V. = 1.79%		97.6 ± 1.75	Mean % recovery ± S.D., C.V. = 1.95%		101 ± 1.99

[a] Average of three readings.

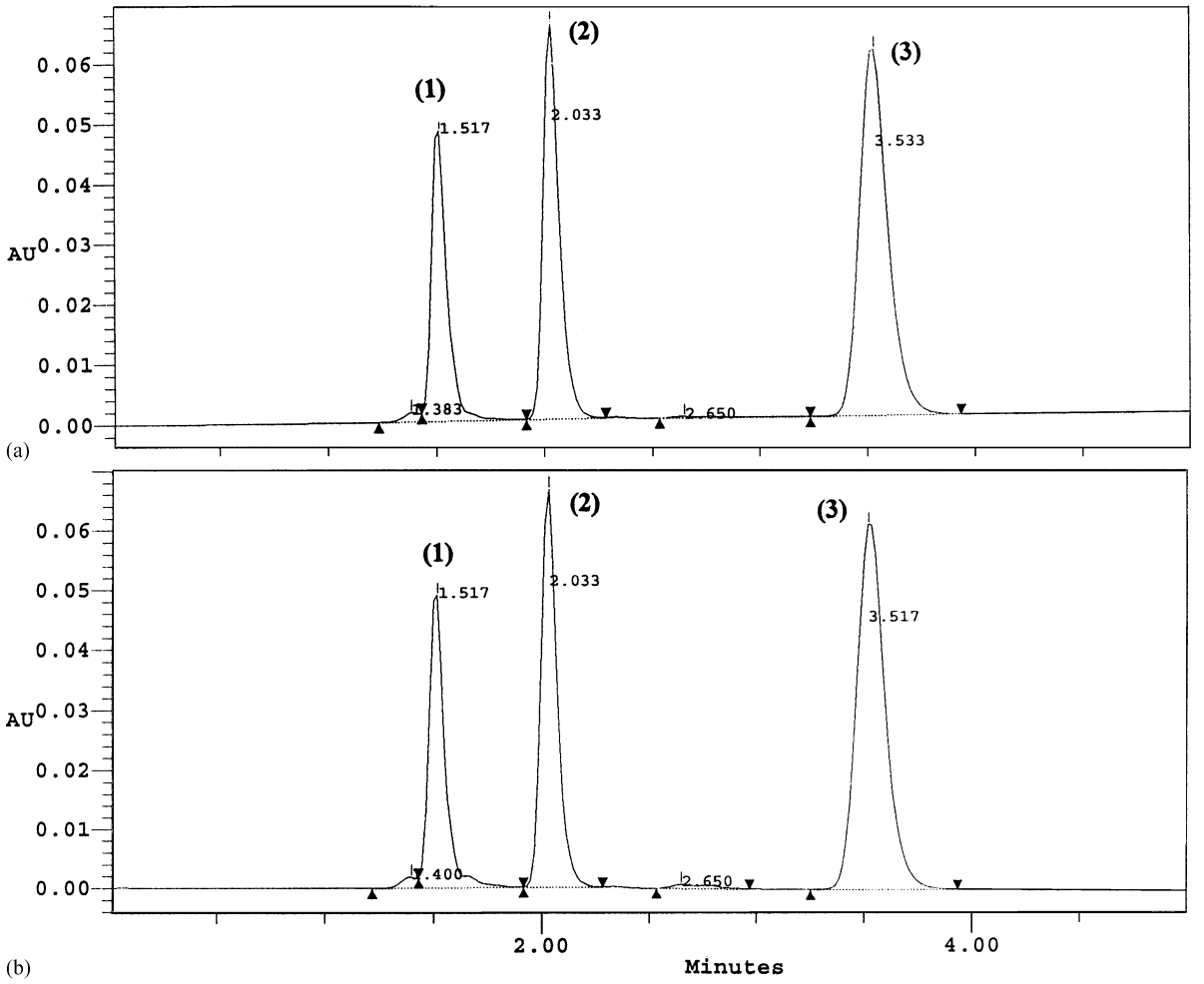

Fig. 3. Typical chromatograms obtained by HPLC analysis of standard (a) and Clarinase® tablets (b), (1) Pseudoephedrine sulfate (480 μg/ml), (2) MPB internal standard (5 μg/ml) and Loratadine (20 μg/ml).

Table 3
Quantitative parameters for the determination of loratadine and pseudoephedrine sulfate with HPLC method

Compound	Linearity range (μg/ml)	Intercept	S.E.M.	Slope	S.E.M.	Correlation coefficient	LOD (μg/ml)
Loratadine	5–100	0.0242	0.0011	0.0679	0.00870	0.9997	0.5
Pseudoephedrine sulfate	120–1200	0.0078	0.0001	0.0016	0.00134	0.9990	60

extract from pharmaceutical tablets (Clarinase®), respectively.

The retention times for [II], internal standard and [I] were found to be 1.5, 2, 3.5 min, respectively. The peak area ratios of pseudoephedrine sulfate to internal standard exhibit linear relationship with their concentrations. The regression equations, correlation coefficient of calibration curves are shown in Table 3.

Table 4
Recovery of loratadine and pseudoephedrine in synthetic mixtures by the proposed HPLC method[a]

Loratadine			Pseudoephedrine sulfate		
Added (μg/ml)	Found (μg/ml)	% Recovery	Added (μg/ml)	Found (μg/ml)	% Recovery
15	14.5	96.9	480	494.4	103.0
20	21.0	105.0	480	495.1	103.2
25	25.3	101.2	480	488.7	101.8
20	21.0	105.0	360	365.1	101.4
20	21.0	105.0	600	601.1	100.2
Mean% recovery ± S.D. C.V. = 2.86		102.6 ± 2.94	Mean% recovery ± S.D. C.V = 1.27		102.0 ± 1.3

[a] Average of three readings.

The recovery test was performed using synthetic mixtures containing various amounts of [I] and [II].The results are reproducible and precise as C.V are very low (Table 4).

The HPLC method was applied for analysis of Clarinase®, Mosedin®, Loratan®, Claritine® tablets in addition to Loratan® syrup and Mosedin® syrups. In the case of syrups; PPB was used as internal standard due to presence of MPB in syrup as preservative (Tables 5 and 6).

The specificity of the method was studied where the mean percentage recovery of [I]and [II] was separately determined in mixtures of them with possible interfering materials such as starch, magnesium stearate, and lactose. The results obtained exhibited no interferences. Furthermore, the chromatograms of standard loratadine and pseudoephedrine mixture in the ratio (1:24) when compared with that of Clarinase® tablets, Fig. 3 showed the same retention times for both compounds which indicates the specificity of the proposed HPLC method.

Table 5
Results of the simultaneous determination of loratadine and pseudoephedrine sulfate in Clarinase® tablets[a]

Drug	% Recovery ± S.D.	
	[1]D spectrophoptometric method	HPLC method[b]
Loratadine	96.3 ± 1.2	96.1 ± 0.21
Pseudoephedrine sulfate	99.1 ± 1.83	98.6 ± 0.90

[a] Average of three readings.
[b] MPB is used as internal standard.

Table 6
Determination of loratadine in different dosage forms using the two proposed methods[a]

Dosage form	% Recovery ± S.D.	
	[1]D spectrophoptometric method	HPLC method[b]
Loratan® tablets	95.5 ± 0.9	95.2 ± 0.36
Claritine® tablets	97.5 ± 0.41	99.4 ± 0.38
Mosedin® tablets	99.5 ± 0.6	99.4 ± 0.62
Loratan® syrup	–	93.4 ± 0.52
Mosedin syrup	–	96.5 ± 0.78

[a] Average of three readings.
[b] PPB is used as internal standard in HPLC method.

The HPLC method was proved to be stability indicating method for loratadine where the stability of loratadine was studied in different pH and different temperatures and the degradation product obtained by the procedures mentioned previously exhibited additional peak in the in the chromatogram at 1.78 min. However, during actual analysis of all samples this peak does not appear which suggests that [I] is stable during the analytical procedures.

4. Conclusion

This work describes two simple methods for simultaneous determination of both drugs without prior separation with the advantage of high

sensitivity of derivative spectrophotometry and the speed of HPLC method where the whole analysis takes not more than 4 min making them more convenient for quality control purposes.

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