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Spectrophotometric Methods for Estimation of Ibuprofen and Pseudoephedrine Hydrochloride in Combined Dosage Form

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Two accurate, economical and reproducible methods for estimation of ibuprofen and pseudoephedrine hydrochloride in combined dosage form have been developed. The first method involves two-wavelength calculation. The two wavelengths selected for estimation of ibuprofen are 264.0 nm and 254.5 nm while those for pseudoephedrine hydrochloride are 257.5 nm and 266.5 nm. The second method is based on quantitative formation of hexane extractable copper(II) complex of ibuprofen which shows maximum absorbance at 697 nm and benzene : carbon disulphide (3 : 1) extractable copper(II) complex of pseudoephedrine hydrochloride which shows maximum absorbance at 437 nm. Both drugs obey Beer's law in the concentration ranges employed for these methods. Results of analysis were validated statistically.

INTRODUCTION

Both ibuprofen and pseudoephedrine hydrochloride are official in USP, BP and IP. For estimation of ibuprofen IP[1] and BP[2] suggest titrimetric method while USP[3] suggests chromatographic method. For pseudoephedrine hydrochloride all three pharmacopoeias suggest chromatographic method of estimation from tablets. Methods reported for estimation of ibuprofen from formulations include spectrophotometric[4–9], chromatographic[10–20] and polarographic[21]. Reported methods for estimation of pseudoephedrine hydrochloride from formulations includes spectrophotometric[22, 23] and chromatographic[24–29]. None of the methods is reported for estimation of both drugs in combined dosage forms.

EXPERIMENTAL

Instrument

Jasco UV/visible recording spectrophotometer (Model-7800) Spectral band width : 3 nm; wavelength accuracy: ±0.5 nm (with auto wavelength correction), 10 nm matched quartz cells.

Reagents and solutions

1. 3% Copper(II) acetate solution in distilled water.
2. Strong ammonia solution

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3. Benzene-carbon disulphide mixture: Mixed 3 volumes of benzene and 1 volume of carbon disulphide.
4. Ethanol
5. Hexane
6. Methanol
7. Distilled water.

All reagents used were of analytical-reagent grade.

Procedure

Method I

Pure drug samples of ibuprofen and pseudoephedrine hydrochloride were dissolved in methanol separately and were scanned from 300 nm to 250 nm. From respective spectra two wavelengths λ_1 (264.0 nm) and λ_2 (254.5 nm) for estimation of ibuprofen and two wavelengths λ_3 (257.5 nm) and λ_4 (266.5 nm) for estimation of pseudoephedrine hydrochloride were selected on the basis of the principle that the absorbance difference between two points on the mixture spectra is directly proportional to concentration of the component of interest and independent of the interfering component. Fig. 1 represents the overlain spectra of ibuprofen and pseudoephedrine hydrochloride in methanol and the method for selection of two wavelengths for both the drugs.

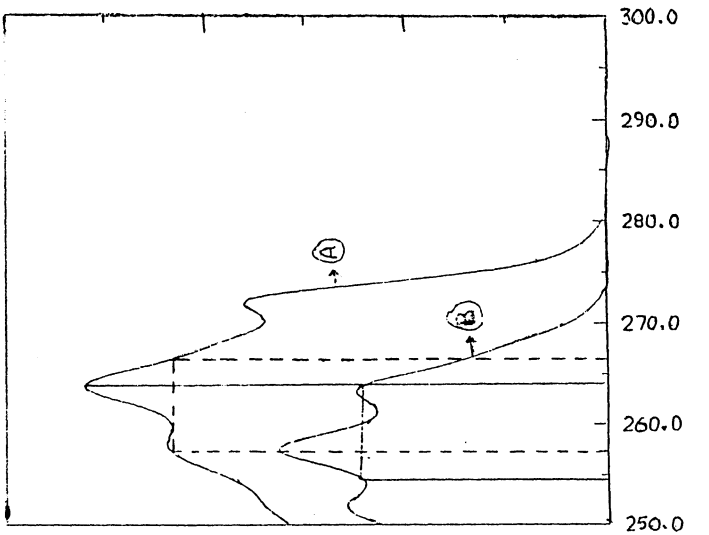

Fig. 1 Overlain spectra of ibuprofen (A) and pseudoephedrin in HCl (B) in methanol for two wavelength method

Nine mixed standards of pure drug containing 0–500 mcg/mL of ibuprofen and pseudoephedrine hydrochloride were prepared in methanol. All standards were scanned at λ_1 and λ_2 for ibuprofen and λ_3 and λ_4 for pseudoephedrine hydrochloride using methanol as blank. Absorbance $(A_1 - A_2)$ values were measured and are reported in Table-1. Respective calibration curves were plotted.

TABLE-1
ABSORBANCE ($A_1 - A_2$) VALUES OF IBUPROFEN AND PSEUDOPHEDRINE HYROCHLORIDE IN MIXED STANDARDS

Standard No.	Conc. of Ibuprofen (mcg/mL)	$A_1 - A_2$	Conc. of pseudoephedrine HCl (mcg/mL)	$A_1 - A_2$
01	0	0.001	0	0.001
02	50	0.020	50	0.046
03	100	0.043	100	0.087
04	150	0.062	150	0.125
05	200	0.080	200	0.170
06	250	0.099	250	0.202
07	300	0.116	300	0.239
08	350	0.128	350	0.281
09	500	0.182	500	0.403

Procedure for tablet formulation

Weighed tablet powder equivalent to 200 mg of ibuprofen was transferred to 100 mL volumetric flask. Accurately weighed 170 mg pure drug sample of pseudoephedrine hydrochloride was transferred to the same volumetric flask. The powder was dissolved in 75 mL of methanol and volume made up to the mark with methanol. The solution was filtered through whatman filter paper no. 41. 1 mL filtrate was diluted to 10 mL with methanol. The solution was analyzed by scanning at respective wavelengths. Thus, the quantity of ibuprofen and pseudoephedrine hydrochloride using respective calibration curves. Results of analysis are reported in Table-2.

TABLE-2
RESULTS OF ANALYSIS OF COMMERCIAL TABLETS

Tablet	Batch I		Batch II	
	PS	IB	PS	IB
Label claim (mg/tab)	30	200	60	400
% of label claim estimated*:				
Method I	99.350	101.250	98.390	98.940
Method II	99.240	99.880	97.780	99.000
S.D.:				
Method I	1.288	2.165	0.571	0.277
Method II	1.139	2.220	0.331	1.530
R.S.D.:				
Method I	1.296	2.138	0.580	0.277
Method II	3.820	1.110	0.565	0.386

*Average of three readings; PS = Pseudoephedrine hydrochloride and IB = Ibuprofen.

Method-II

Calibration curve of Ibuprofen

Accurately weighed 500 mg ibuprofen was dissolved in 50 mL of ethanol so as to give stock solution of concentration 10 mg/mL. Stock solution was diluted with ethanol to give 7 dilutions of concentration ranging from 0–6 mg/mL of ibuprofen. To 5 mL of each diluted standard taken in a separating funnel, 3 mL of 3% aqueous solution of copper(II) acetate was added. The reaction mixture was gently shaken for 5 min. This solution was then extracted with 5, 3 and 2 mL of hexane. Combined the hexane layer and measured the absorbance of hexane layer at 697 nm against reagent blank. Calibration curve was plotted. Beer's law was obeyed in concentration range of 0–8 mg/mL of ibuprofen.

Calibration curve of Pseudoephedrine Hydrochloride

Accurately weighed 100 mg pseudoephedrine hydrochloride was dissolved in 50 mL ethanol so as to give stock solution of concentration 2 mg/mL. Stock solution was diluted with ethanol to give 7 dilutions of concentration ranging from 0–600 mcg/mL of pseudoephedrine hydrochloride. 5 mL of each dilution was treated similar to that for calibration curve for ibuprofen up to extraction with hexane. Hexane layer was discarded and aqueous layer was heated on water bath at 75°C for 20 min. To the resulting solution in each dilution was added 2 mL of strong ammonia solution, shaken and extracted with 10 mL of benzene : carbon disulphide mixture (3 : 1). Collected organic layer in a stoppered flask after filtration through Whatmen filter paper no. 41 and measured absorbance of organic layer at 437 nm using reagent blank. Calibration curve was plotted. Beer's law was obeyed in the concentration range of 0–600 mcg/mL of pseudoephedrine hydrochloride.

Procedure for tablet formulation

Weighed tablet powder equivalent to 100 mg of ibuprofen was transferred to a 50 mL volumetric flask, added 30 mL of ethanol and filtered through Whatman filter paper no. 41 into another 50 mL volumetric flask. Washed filter paper with ethanol and collected the washing in the same volumetric flask. Finally, made up the volume to 50 mL mark with ethanol.

5 mL of this solution was treated as per the procedure given for calibration curve of ibuprofen. Collected the hexane layer which was used for estimation of the quantity of ibuprofen by measuring absorbance at 697 nm. Aqueous layer was treated as per the procedure given for calibration curve of pseudoephedrine hydrochloride. Collected the benzene : carbon disulphide layer which was used for estimation of quantity of pseudoephedrine hydrochloride by measuring absorbance at 437 nm. Concentration of both the components was determined using respective calibration curve. The analysis was repeated 3 times for two different strengths of tablets. Results of analysis are tabulated in Table-2. Overlain spectra of ibuprofen-copper complex in hexane and pseudoephedrine hydrochloride-copper complex in benzene : carbon disulphide are reported in Fig. 2.

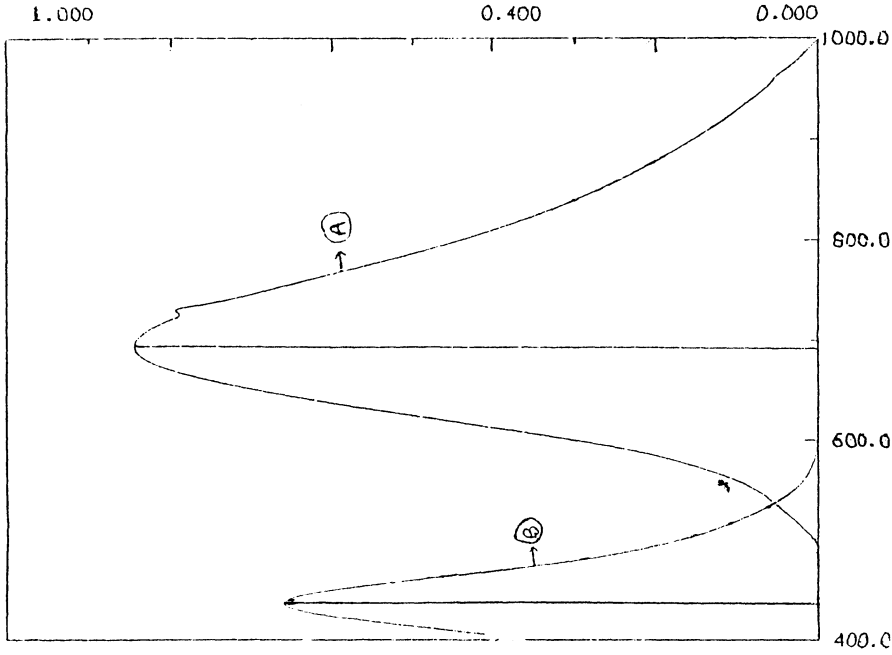

Fig. 2 Overlain spectra of ibuprofen-copper complex (A) and pseudoephedrine-Copper complex (B).

RESULTS AND DISCUSSION

The proposed methods for estimation of ibuprofen and pseudoephedrine hydrochloride in combined dosage form were found to be accurate, simple and reproducible. The first method employing two-wavelength calculation can be used for simultaneous analysis of two drugs in combined dosage forms. Selection of two wavelengths totally eliminates the spectral interference from one of the two drugs while estimating the other. Since the concentration of pseudoephedrine hydrochloride in tablet sample was much less in comparison to that of ibuprofen so standard addition method was used. The second method developed can also be used for estimation of the two drugs in combined dosage form. This method is more complicated and time consuming than method I but has the advantage that it can be used with any model of spectrophotometer with reproducible resutls.

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