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Estimation of Atenolol and Nifedipine in Multicomponent Formulations by Ultraviolet Spectroscopy

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Spectrophotometric methods requiring no prior separation have been proposed for simultaneous estimation of atenolol and nifedipine in two component tablet and capsule formulations. The methods employ first derivative spectrophotometry and ratio compensation technique for the simultaneous analysis of the two drugs. The absorbance maxima of atenolol was found to be at 276 nm and that of nifedipine was at 340 nm, in 50% v/v aqueous methanol. Atenolol and nifedipine obeyed Beer's law in the concentration ranges employed for these methods. Results of analysis and recovery studies were statistically evaluated for their accuracy and precision.

INTRODUCTION

Atenolol, chemically 2-[4-(2-hydroxy-3-isopropylaminopropoxy)-phenyl] acetamide is a beta-adrenoceptor blocker. IP[1] and BP[2] describe non-aqueous titrimetric assay procedure for the bulk drug and UV spectrophotometric assay procedure for tablets. Nifedipine, chemically 1,4-dihydro-2,6-dimethyl-4-(2-nitrophenyl) pyridine-3,5-dicarboxylate is an antianginal vasodilator and used in the management of a variety of cardiovascular disorders. IP[1] and BP[2] describe titrimetric assay procedure for bulk drug and UV spectrophotometric assay procedure has been reported in IP for capsules and tablets. A HPLC method is described in USP[3] for the analysis of nifedipine capsules.

Various reported methods include spectrophotometric[4–6], HPLC[7,8], TLC[9,10] and gas chromatographic[11,12] techniques for the estimation of atenolol and nifedipine in dosage forms and biological fluids. But none of the methods described their simultaneous spectrophotometric estimation from combined dosage forms.

EXPERIMENTAL

A Shimadzu UV/Visible recording spectrophotometer (Model 160A) was used for spectral measurement in 10 mm matched quartz cells. Instrumental parameters were: spectral band width 2 nm, wavelength accuracy ± 0.5 nm with automatic wavelength correction.

Methanol (50% v/v) was used as a solvent. The analysis was carried out under protected conditions from light to prevent photodegradation of nifedipine.

Procedure

Method I: Employing Ratio Compensation Technique

Standard stock solution of strength 200 mcg/mL and of atenolol and nifedipine were made in 50% v/v aqueous methanol separately. The overlain spectra of atenolol (200 mcg/mL) and nifedipine (40 mcg/mL) is given in Fig. 1.

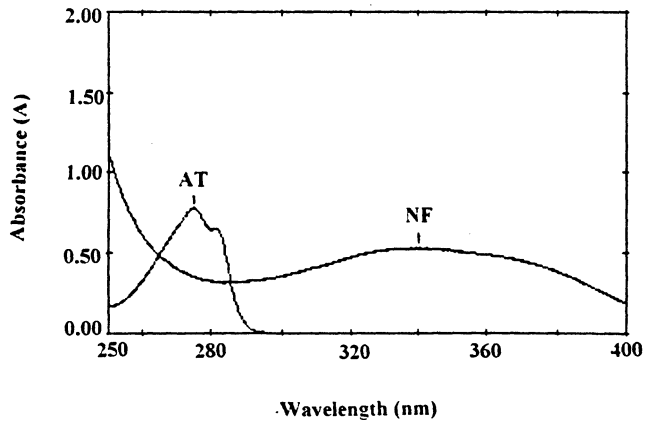

Fig. 1 Overlain spectra of atenolol (AT) and nifedipine (NF)

The E (1%, 1 cm) values of both the drugs were determined at 340 nm and 276 nm. Calibration curve for nifedipine was plotted in the concentration range of 0–50 mcg/mL using absorbance values at 340 nm. Concentration of nifedipine in the sample solution was determined directly from the absorbance at 340 nm, where atenolol showed no absorbance. Calibration curve for atenolol was plotted in the concentration range of 0–100 mcg/mL using absorbance values at 276 nm. Concentration of atenolol in the sample solutions was determined by calculating the absorbance contributed by atenolol to the absorbance at 276 nm. The absorbance contribution of atenolol was calculated from the equation given below:

$$AB = \frac{AA}{1.1453}$$

where AA = Absorbance of the sample solution at 340 nm

AB = Absorbance of the sample solution at 276 nm.

Preparation and Analysis of Tablet Sample Solutions

Twenty tablets were weighed and ground to fine powder. An accurately weighed quantity of the powder equivalent to 10 mg of nifedipine was transferred to 100 mL volumetric flask. It was dissolved with the aid of 50 mL of methanol and volume was made up to the mark with distilled water. The solution was

filtered through Whatman filter paper No. 41 and the solution was further diluted to get the final concentration of 100 mcg/mL of atenolol and 40 mcg/mL of nifedipine. The solution obtained was analysed by recording the absorbance at 340 nm and 276 nm. The concentration of atenolol in the sample solution was determined using the above equation and the concentration of nifedipine was determined directly from the absorbance at 340 nm, where atenolol show no absorbance. The results of analysis of tablet formulation are stated in Table-1.

TABLE-1
RESULTS OF ANALYSIS OF COMMERCIAL TABLET AND CAPSULE FORMULATIONS

Commercial samples	Label claim mg/tab or mg/cap		% of label claim estimated*				Standard deviation			
			Method-I		Method-II		Method-I		Method-II	
	AT	NF	AT	NF	AT	NF	AT	NF	AT	NF
Tablet	50	20	102.88	100.65	101.90	102.15	2.07	1.56	1.31	1.18
Capsule A	50	20	99.12	97.38	102.15	98.76	1.71	1.98	2.32	0.98
Capsule B	50	10	102.32	101.30	101.36	99.27	2.19	0.76	1.76	1.21

*Average of five estimations; AT = Atenolol; NF = Nifedipine

Preparation and Analysis of Capsule Sample Solutions

Twenty capsules belonging to the same batch were emptied, weighed and the contents were powdered and mixed. An accurately weighed powder sample equivalent to 10 mg of nifedipine was transferred to a 100 mL volumetric flask. The powder mixture was dissolved in 50 mL of methanol and the volume was made up to the mark with distilled water. The solution was filtered through Whatman filter paper No. 41 and was further diluted to get 100 mcg/mL of atenolol and 40 mcg/mL of nifedipine for formulation A and 100 mcg/mL of atenolol and 20 mcg/mL of nifedipine for formulation B. The results of analysis of capsule formulation are given in Table-1.

The recovery studies conducted by addition of different amount of pure drugs to a pre analysed tablet/capsule sample solution gave satisfactory recovery data in the range of 98–103%.

Method II: Employing first derivative spectrophotometry

From the first derivative spectra of the two drugs (Fig. 2) It was evident that atenolol and nifedipine show zero absorbance at 314 nm and 287 nm respectivley. As at the zero crossing point on the first derivative spectra of one drug the other drug shows substantial absorbance, these two wavelengths can be employed for the estimation of nifedipine and atenolol respectively without any interference from the other drug in their combined formulation. Six mixed standards having concentrations 0, 20, 40, 60, 80 and 100 mcg/mL of atenolol and 50, 40, 30, 20, 10 and 0 mcg/mL of nifedipine respectively were prepared by using the appropriate volume of standard stock solutions. All the mixed standard solutions

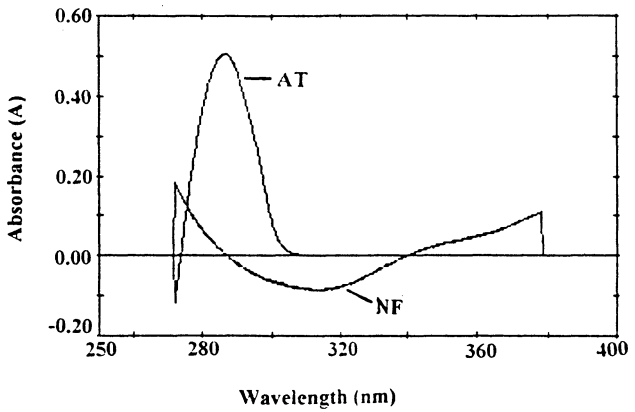

Fig. 2. First order derivative spectra of atenolol (AT) and nifedipine (NF)

were scanned over the range of 400–250 nm. The resultant spectra were derivatised to obtain first derivative order spectra. The absorbances were measured for atenolol at 287 nm (zero crossing of nifedipine) and for nifedipine at 314 nm. These absorbances were plotted against concentration to obtain calibration curve.

Tablet and capsule sample solutions were made as mentioned in method I. Absorbances of these sample solutions were recorded at 287 nm and 314 nm from the first derivative spectra of the sample solution and the amount of drug present in the sample solution was obtained from the calibration curves plotted. The results of analysis using this method are given in Table-1. The recovery studies carried out gave satisfactory results in the range of 99–102%.

RESULTS AND DISCUSSION

The proposed methods for simultaneous estimation of atenolol and nifedipine in combined dosage forms were found to be accurate, simple, rapid and can be used for the routine analysis of atenolol-nefedipine combination. The values of standard deviation were low and recovery was close to 100% suggesting satisfactory accuracy and reproducibility of the method.

The first method employing ratio compensation technique is a very simple method which can be employed for analysis of both drugs in combined dosage forms using the simplest form of the instrument. After recording the absorbances at the selected two wavelengths the concentration of both the drugs can be found out by simple calculations.

The second method requires spectral data processing and hence can be applied only on recording spectrophotometers. The first derivative spectrum was found to eliminate the spectral interference from one drug while estimating other drug and vice-versa.

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