

## KINETIC SPECTROPHOTOMETRIC DETERMINATION OF ATENOLOL IN PERCHLORIC ACID MEDIUM

RASHMI AGARWAL<sup>1\*</sup> AND ANAND G FADNIS<sup>2</sup>

<sup>1</sup>Department of Pharmaceutical Chemistry, Govt. Holkar Science College, Indore 452017 and <sup>2</sup>SCM Institute of Professional Studies, 70 Ganesh Ganj, Indore 452002. \*Email: rasha\_75@rediffmail.com

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### ABSTRACT

A simple, rapid and sensitive spectrophotometric method has been proposed for the determination of atenolol using cerium (IV) in perchloric acid medium. The method is based on measuring the decrease in absorbance of cerium(IV). The fixed time and fixed absorbance method were used for the quantitative determination of atenolol employing Ceric sulphate as an oxidant in 0.7M Perchloric acid. The developed method can be successfully used for the determination of atenolol in bulk drug and in tablets without any interference of common excipients.

**Keywords:** Spectrophotometric, Kinetic determination, Atenolol, Perchloric acid.

### INTRODUCTION

Atenolol, chemically known as 4-(2-hydroxy-3-isopropylaminopropoxy)Phenyl-acetamide. Atenolol(ATN) is a  $\beta_1$ -selective (cardioselective)  $\beta$ -adrenergic receptor-blocking agent without membrane stabilizing or intrinsic sympathomimetic (Partial agonist)

activities<sup>1-2</sup>. ATN is also used to treat myocardial infarction (heart attack), arrhythmias (rhythm disorders), angina (chest pain), and disorders arising from decreased circulation and vascular constriction, including migraine. In the European pharmacopoeia<sup>3</sup>, the described method for atenolol quantification uses technique of titration with acid perchloric. In the British Pharmacopoeia<sup>4</sup> the indicated methods use spectrophotometric in the ultraviolet at 275nm. Metal ion oxidants have been widely employed in synthetic chemistry<sup>5-7</sup> including kinetic cerimetric estimations of various aldoses<sup>8</sup>. These are stable, inexpensive and can readily be stored and handled. Kinetic method of analysis have been widely developed and accepted in chemical analysis of different samples<sup>9-11</sup>. The basic advantage of cerium (IV) over other oxidants is its reduction to a single substance, cerium (III), without any intermediate reactions. The present work describes the procedure for the estimation of Atenolol using kinetic rate data obtained from redox indicator reaction between atenolol with cerium(IV) in acidic medium whose kinetic and mechanistic features has been elaborated in earlier studies.

### MATERIALS AND METHODS

#### Material

Commercially available chemicals of pure quality were used without further purification. A stock solution of atenolol (IPCA Laboratories Ltd., Ratlam) was prepared by dissolving appropriate amount of sample in double distilled water. Ce (IV) stock solution was prepared by dissolving ceric sulphate (99.9% Loba chem.) in aqueous sulphuric acid.

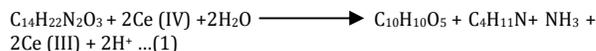
#### Kinetic method

The kinetic runs were performed in stoppered glass vessel in a controlled temperature ( $\pm 0.1^\circ\text{C}$ ) water bath. The kinetics of redox reaction between atenolol and cerium (IV) in Perchloric acid medium was followed under pseudo first order condition in presence of excess concentrations of atenolol by measuring the absorbance at 360nm for Ce(IV)-Visible spectrophotometer Systronics-104 at constant temperature. The unknown is calculated in simulated sample by fixed time method and fixed absorbance method from the regression equations and from the graphs.

### RESULTS AND DISCUSSION

#### Indicator reaction

The reaction followed first order kinetics. The indicator reaction between atenolol and cerium (IV) in Perchloric acid medium under the kinetic run condition can be represented as



#### Kinetic Estimation

The rate of these indicator reactions under the present kinetic conditions in terms of disappearance of metal ion concentration with time at constant temperature and perchloric acid concentration can be given by

$$-\frac{d[\text{Ce}^{\text{IV}}]}{dt} = k_0[\text{Cerium(IV)}]_0[\text{atenolol}]_0 = k_{\text{obs}}[\text{Ce}^{\text{IV}}]_0 \dots (2)$$

Thus when  $[\text{atenolol}]_0 \gg [\text{Ce}^{\text{IV}}]_0$  then,

$$k_{\text{obs}} = k_0[\text{atenolol}] \dots (3)$$

Equation (2) alternatively expressed as eq.(4) for the finite change in the initial concentration of cerium(IV) in a given fixed time interval.

$$-\frac{\Delta[\text{Ce}^{\text{IV}}]}{\Delta t} = k_0[\text{Ce}^{\text{IV}}]_0[\text{atenolol}]_0 \dots (4)$$

The initial concentration of cerium (IV) i.e.  $[\text{Ce}^{\text{IV}}]_0$  is kept constant in each kinetic run varying initial concentration of atenolol i.e.  $[\text{atenolol}]_0$  therefore,

$$-\frac{\Delta[\text{Ce}^{\text{IV}}]}{\Delta t} = k_0[\text{atenolol}]_0 \dots (5)$$

Eq.(3) and (5) have been used to obtain three different calibration plots to determine the concentration of simulated samples of atenolol.

The kinetic data of variation of  $k_{\text{obs}}$  [Fig.1], absorbance at fixed time interval [Fig.2] and time at fixed absorbance with initial concentration of atenolol [Fig.3] in different kinetic runs are given graphically for atenolol.

The data of table-1 has been used to obtain the following regression equations for the three linear calibration plots to obtain unknown concentration of simulated samples of atenolol. The results of these estimations with actual theoretical values are given in the table-1.

Table 1: Variation of  $k_{obs}$  absorbance at fixed time and time at fixed absorbance with initial Concentration of Atenolol.

[Atenolol] X10 <sup>3</sup> (Sec)(Moldm <sup>-3</sup> )	$k_{obs} \times 10^4 (S^{-1})$	Absorbance at fixed 10 <sup>-2</sup> time			10 <sup>-2</sup> time (Sec) at Fixed absorbance		
		2.4	4.8	7.2	0.7	0.5	0.3
2.5	18.4	0.664	0.432	0.268	2.1	3.7	6.7
3.125	20.6	0.627	0.386	0.236	1.8	3.6	5.9
4.375	26.3	0.543	0.292	0.154	1.4	2.5	4.7
5	29.3	0.512	0.251	0.126	1.3	2.4	4.2
5.625	32.2	0.472	0.219	0.103	1.2	2.2	3.7
6.875	36.4	0.431	0.179	0.077	1	1.9	3.5
Unknown-1	23.6	0.589	0.342	0.198	1.7	3.1	5.4
10 <sup>3</sup> [Unknown-1] moldm <sup>-3</sup>	33.72		3.76	3.76	3.71	3.72	3.77
Unknown-2	34.3	0.452	0.194	0.083	1.1	2	3.6
10 <sup>3</sup> [Unknown-2] moldm <sup>-3</sup>	36.21		6.23	6.26	6.2	6.2	6.18

10<sup>4</sup>[Ce(IV)]=2.5moldm<sup>-3</sup>, [HClO<sub>4</sub>]=0.7moldm<sup>-3</sup>, Temperature=296±0.1K, λ=360nm

**RESULTS**

Rate Constant Method

10<sup>3</sup>[Unknown-1]moldm<sup>-3</sup> = 3.73±0.02 (calculated): 3.75(actual)

10<sup>3</sup>[Unknown-2]moldm<sup>-3</sup> = 6.25 (calculated): 6.25(actual)

Fixed Time Method

10<sup>3</sup>[Unknown-1]moldm<sup>-3</sup> = 3.74±0.01 (calculated): 3.75(actual)

10<sup>3</sup>[Unknown-2]moldm<sup>-3</sup> = 6.23 ± 0.02(calculated): 6.25(actual)

Fixed Absorbance Method

10<sup>3</sup>[Unknown-1]moldm<sup>-3</sup> = 3.73 ± 0.02 (calculated): 3.75(actual)

10<sup>3</sup>[Unknown-1]moldm<sup>-3</sup> = 6.22 ± 0.03 (calculated): 6.25(actual)

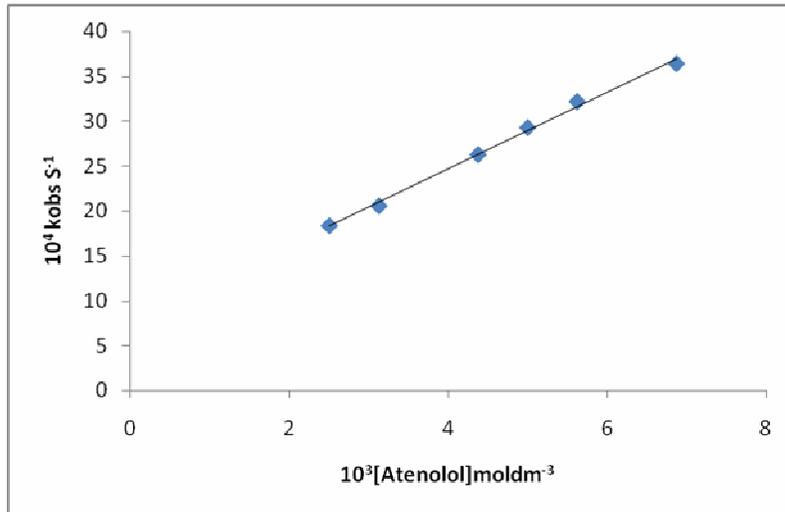


Fig. 1: Estimation of atenolol by variation of  $k_{obs}$  with [atenolol]. 10<sup>4</sup>[Ce<sup>IV</sup>] = 2.5moldm<sup>-3</sup>; [HClO<sub>4</sub>] = 0.7moldm<sup>-3</sup>; Temp=296K; λ=360nm.

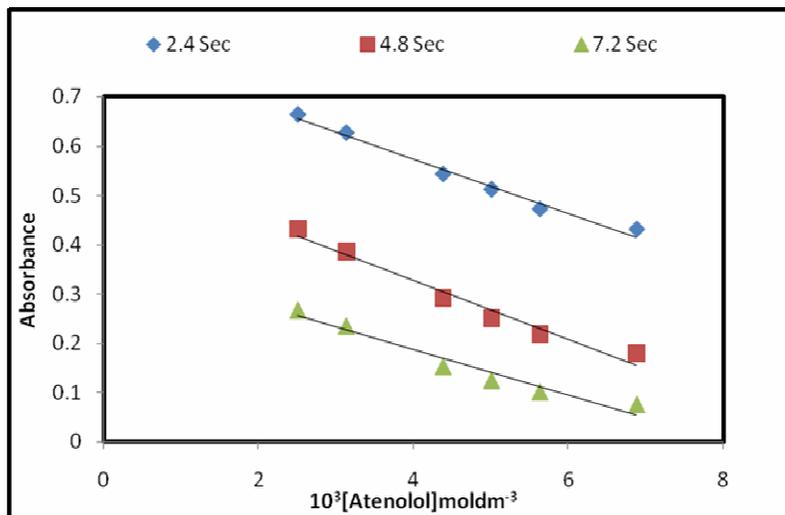


Fig. 2: Estimation of atenolol by variation of absorbance at fixed time. 10<sup>4</sup>[Ce<sup>IV</sup>]=2.5moldm<sup>-3</sup>; [HClO<sub>4</sub>] = 0.7moldm<sup>-3</sup>; Temp=296K; λ=360nm.

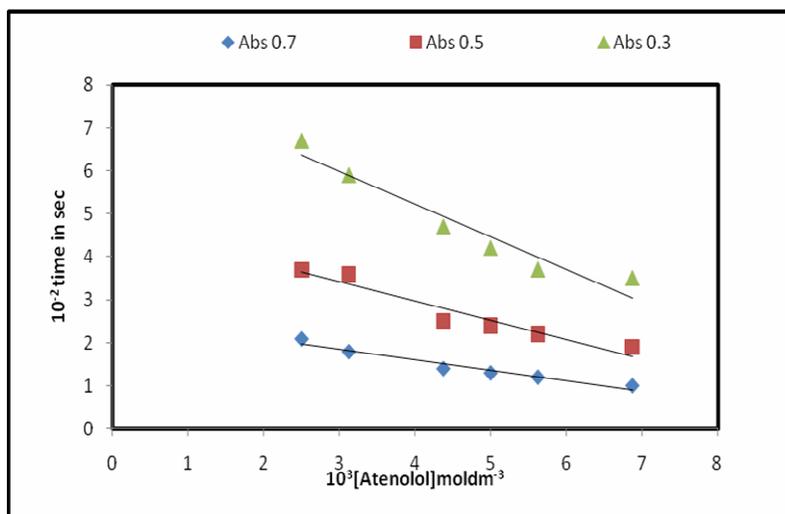


Fig. 3: Estimation of atenolol by variation of time at fixed absorbance.  $10^4[\text{Ce}^{IV}] = 2.5 \text{ mol dm}^{-3}$ ;  $[\text{HClO}_4] = 0.7 \text{ mol dm}^{-3}$ ;  $\text{Temp} = 296\text{K}$ ;  $\lambda = 360\text{nm}$ .

### CONCLUSION

The visible spectroscopy method demonstrated here is applicable to the estimation of atenolol in pure as well as dosage forms. The experiments have been performed on calibrated equipments using suitable reference standards. The proposed spectrophotometric estimation<sup>12-14</sup> method is found to be simple, sensitive, accurate, precise & economical and can be used in the determination of atenolol in bulk drug and its pharmaceutical dosage forms in a routine manner.

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