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Spectrophotometric Determination of Metoprolol and Atenolol by Iron (III) and Ferricyanide

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Abstract
Easy, accurate, sensitive method used to determine of metoprolol and atenolol both in pure form. The work was on reducing Fe (III) in acid medium & following reaction of Fe (II) with ferricyanide to form Prussian blue. That has a maximum absorption at λmax 762 nm in both cases. Beer's law in the concentration ranges 5.0 – 16.0 & 1.6 – 5.0 mg / ml, for metoprolol & atenolol The molar absorptivity are 1.74 x 10^4 and 4.22 x 10^4 L /mol.cm, sandal sensitivity are 23.15 & 8.06 mg/cm^2.the limits of detection quantification are reported . 7 repeat analyses of solutions having 3 different concentrations of each drug were passed & the percent error and the RSD values have been informed.

Keywords: metoprolol; Atenolol; Spectrophotometric; Ferricyanide

Introduction

Metoprolol, 1-(isopropyl amino)-3-[p-(2-methoxyethyl) phenoxy]-2- propanol, is a kind of adrenaline receptor blocker. It is widely used for the treatment of hypertension, angina, myocardial Infarction, arrhythmia,
hyperthyroidism & other related diseases [1, 2]. The determination of metoprolol occure by several methods involving high- performance liquid (HPLC) [6–10], gas chromatography – mass spectrometry (GC – MS) [3-5]
LC–MS–MS [14] and LC–MS [11–13] in human plasma & extra Biological fluids. ß-Blockers contain analogous chemical structures with highly polar functional groups that make them unsuitable for analysis by GC Methods.
Newly the usage of mass selective detectors using a capillary GC attached
to mass spectrometry as a mode of detection has significantly increased. Suitable derivatization should progress the Gas chromatographic properties of the compounds and produced compounds & high relative
intensity [15]. Atenolol, is a cardio selective β-blocker antagonist, chemically identified as 2-{4-[2-hydroxy-3-(propan-2-ylamino) propoxy] phenyl} acetamide [16].

Atenolol is taked to treat high blood. pressure (hypertension) (HTN) and chest pain (angina). Hypertension is also taked to reduction the severity of heart dosage forms because of the great use of drug .numerous analytical methods have been reported which are depended on the bromination reaction of Atenolol with a identified excess of bromate-bromide mixture in acid medium [20, 21] or depended on the oxidation of the Atenolol by a identified excess of ceric (IV) in acid medium [22], or titration of the drug in glacial CH3COOH with acetous per chloric acid to the see end point using crystal violet as indicator to explain the end point [23].UV spectrophotometric methods having sensitive titrimetric used for determination of Atenolol in tablet formulation [24].
The structures of metoprolol & atenolol in Fig. 1.attacks & regulator of some forms of cardiac arrhythmia & for the management of HTN (hypertension) prevention of chest pain (angaina) it may be used only or along with other antihypertensive agents involving thiazidetype diuretics,
hydralazine, prazosin & α-methyldopa [17-19]. It is essential to develop analytical methods for the determination of drug in commercial because of the using of drug is great.

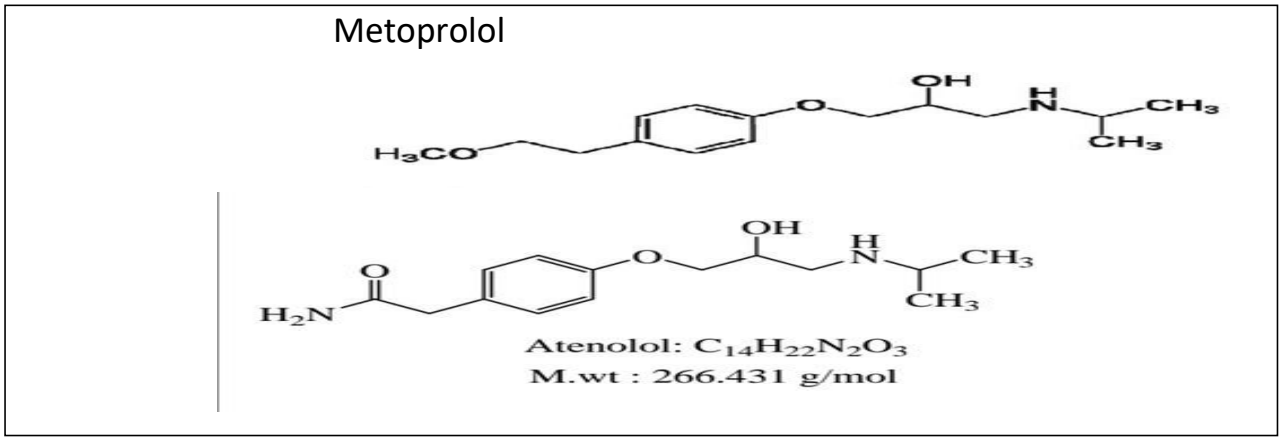

Fig. 1. Structures of metoprolol and atenolol.

Experimental Apparatus:
The measurement of absorbance involved use A Systronics model 107 digital spectrophotometer (India) with 1cm glass cells.

Reagents & solutions:

The preparing all solutions occure by using the analytical reagent and distilled water (H_2O) 0.3% (w/v) solu. Of every anhydrous $FeCl_3$, and the prepared of $K_3Fe(CN)_6$, was occured in water. H_2SO_4 (10 M) was produced by adding 555 ml of H_2SO_4, Sp. Gr. 1.83, which that added slowly to 445 ml of H_2O.

Standard solutions:
Stock solu of metoprolol & atenolol (200 mg/ml) (which is stock solutions) were produced by dissolving process that occurred by dissolve 20 mg of metoprolol and atenolol in ethanol (96%) and by using water 100 ml in order to diluting and the diluting process occure in a flask. The dilution of solution. occurred in order to get standards of 50 metoprolol & 20 mg/ml atenolol with H_2O.

Procedure: Into a series of 10 ml volumetric flasks, (1.0_ 4.0 ml) of metoprolol solu. (50 mg/ ml) & 0.76_ 2.5 ml of 20 mg/ml of atenolol solu. The total volume. Was completed to 3.0 ml by H_2O. And 2 ml of $FeCl_3$ (0.2%) & ferricyanide (0.2%) were putted in each flask, mix, 1 ml of 10 M H_2SO_4 was putted in each flask & completed to mark with H_2O. The absorb. Of the produced solu. Was recorded at 762 nm.

Pharmaceutical preparations:
20 tablets were balanced & converted into powder. An amount of the powder equal to 20 mg of metoprolol & atenolol balanced to 100 ml flask. Approximately 30 ml H_2O having 10 ml of ethanol (96%) were added. &. Completed to the mark with H_2O mix. The filtrate (200 mg/ml) was diluted to produce 50 mg/ml of metoprolol or 20 mg/ml of atenolol, with H_2O.

Absorption spectra:
Fig2. Greenish that produces from the studied antihypertensive drugs displays absorption at 762 nm, & the particular blanks show absor. At this λmax only. Neither Fe (III) nor ferricyanide solution can absorb at this Wavelength.

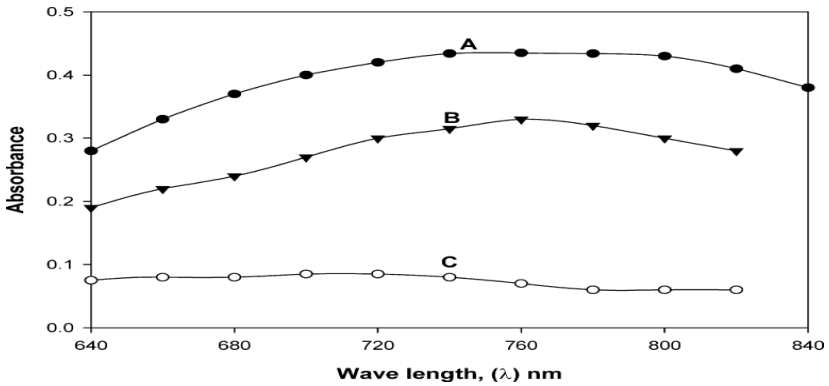

Fig. 2. (A) Reaction metoprolol (100 mg) with Fe (III) & ferricyanide; (B) reaction atenolol (30 mg) with Fe (III)/ferricyanide; (C) blank solution

Optimum Fe (III) & ferricyanide concentrations:
In both cases drugs (metoprolol and atenolol) the absorbance. increased with increase in the volume of 0.2% iron (III) solution & reached maximum when 1.5 ml of the reagent solution were added to 100 mg of metoprolol or 30 mg of atenolol, & 2 ml of 0.2% ferricyanide solution in a total volume of 10 ml. These results indicate that a maximum absorb. Is obtained when the final Fecl (III) conc. is 0.03%. Larger volumes of Fecl (III) up to 2.5 ml had no effect on the sensitivity of the Reaction (Fig. 3). Similar observations were made when varying volumes of 0.2% ferricyanide solution were added to fixed amounts of drug (100 mg of metoprolol or 30 mg atenolol) & Fecl (III) (2 ml; 0.2%) & diluted to 10 ml (Fig. 4).

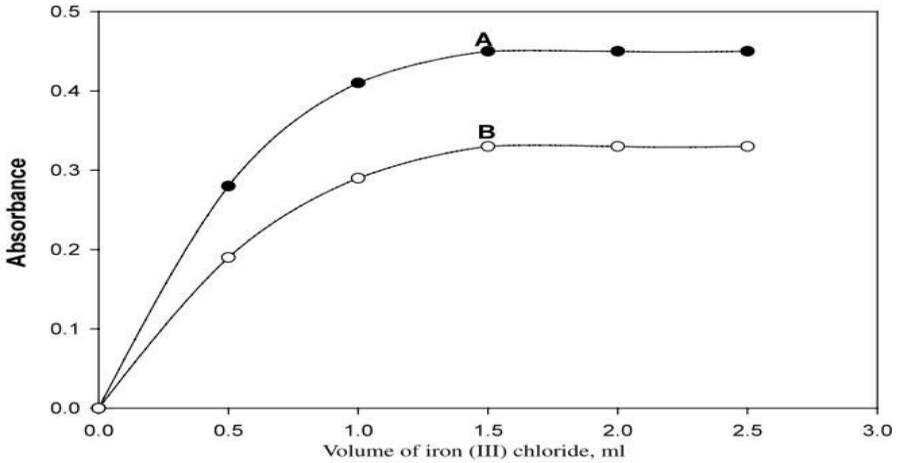

Fig. 3. (A) Metoprolol (100 mg) + 2.0 ml of 0.2% ferricyanide +1.0 ml of 10 M H_2SO_4; (B) Atenolol (30 mg) +2.0 ml of 2% ferricyanide +1.0 ml of 10 M H_2SO_4

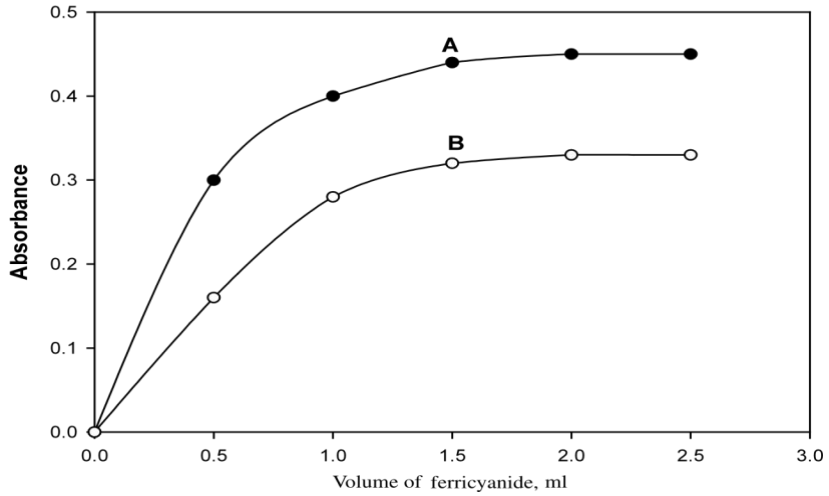

Fig. 4. (A) Metoprolol (100 mg) +2.0 ml of 0.2% Fecl (III) +1.0 ml of 10 M H_2SO_4. (B) Atenolol (30 mg) + 2.0 ml of 0.2% Fecl (III) +1.0 ml of 10 M H_2SO_4.

Analytical appraisal:
Beer's law is obeyed over the concentration ranges, 5.00 – 15.00 & 1.50 – 5.00 mg/ml, for metoprolol & atenolol The molar absorptivities at 762 nm are 1.75 x 10 4 and 4.25 x 104 l/mol cm & Sandal sensitivities are 23.17 & 9.07 mg/cm2 for metoprolol & atenolol, The limits of detection and quantification for metoprolol are 0.14 & 0.44 mg/ml . Similar parameters for atenolol are 0.06 and, 0.18 mg/ml.

Accuracy of method
The method was accurate by executing 7 repeated readings containing different amounts within the limits of Beer's law, percentage error, SD & (RSD) (%) for 7 determinations at each level are shown in Table 1. The accuracy of the method is evident from the percentage error of between 0.68 and 0.17% for metoprolol, and 0.53 and 0.30% for atenolol. RSD values below 3% (Excluding metoprolol, 50 mg level) for 3 different levels studied for both drugs. To evaluate the significance of the results obtained for bulk drug using the proposed procedure, a comparison of the experimental mean values (-x) was made with the true values (m) using n- & t-values. The actual difference between the mean & the true value ($\bar{x}$ - m) was compared with the largest difference that could be expected as a result of indeterminate error (± ts/√n) given in the last Column of Table 1. It is clear from the results that the values of ($\bar{x}$ - m) are less than ± ts= √n indicating no significant difference between the mean & the truevalues.

Table 1. accuracy of method

Drug studied	Amount Taken (mg)	Amount found [a] (mg)	Range (mg)	Error (%)	SD (mg)	RSD (%)	x^- - m	±ts/√n[b]
metoprolol	50.0	44.61	1.47	0.69	2.46	4.91	0.34	2.26
	80.0	81.16	0.81	0.21	0.33	0.41	0.17	0.30
	120.0	120.21	1.20	0.18	0.43	0.35	0.21	0.39
Atenolol	15.0	15.08	1.02	0.54	0.44	2.85	0.08	0.41
	30.0	30.14	1.72	0.44	0.19	0.59	0.14	0.18
	40.0	40.13	0.82	0.31	0.33	0.82	0.13	0.31

Application:
The method was applied to the determination of metoprolol &atenolol in proprietary drugs purchased from local stores & containing other inactive ingredients. The results in Table 2 show that the method is successful for the determination of metoprolol and atenolol and that the excipients in the dosage forms do not interfere. A statistical comparison of results of determination metoprolol and atenolol by the proposed method & reference methods [25, 41] for the same batch of material is presented in Table 3. The Student's t- & F-values.

Table 2. Results of determination of metoprolol and atenolol in dosage forms

Drug	Nominal amount (mg)	Drug found (mg)	(Error)%
(Metoprolol) Lopressor	2.5	2.52	0.80
	5.0	5.06	1.20
	10.0	10.15	1.50
(Atenolol) Vascoten tablet 100mg	2.5	2.54	1.60
	5.0	4.95	1.20
	10.0	9.89	1.50

Indicate that there is no significant difference between the methods in respect of accuracy and precision. To study the reliability & reproducibility of method. The total concentration was found by the proposed method. The determine with each conc. was repeated 3 times & the percent recovery of the added standard was calculated from: % Recovery = $\{C_V - C_u) / C_a\}$ x 100 Where C_V is the total conc. of the analyte measure. C_u, conc. of the analyte presents in the formulation; C_a, conc. of analyte (pure drug) added to formulation. Results of this study present. In Table 4 reveal that the method was unaffected by the various excipients present in the formulations.

Table 3. Comparison of results of metoprolol & atenolol determination by the proposed method with those of reference method

	Proposed method	method		(6.39)
Lopressor(mg)	100.80±0.20	100.40±0.40	2.11	4.00
	101.20±0.21	101.00±0.46	0.94	4.80
	101.50±0.09	101.60±0.10	1.66	1.23
Vascoten(mg)	100.60±0.91	99.50±0.45	2.56	4.09
2.5	99.60±0.35	99.80±0.15	0.45	5.44
5.0	99.50±1.00	100.50±0.85	1.71	1.38
10.0				

Table 4. Results of recovery study by standard-addition method

Formulation	Amount of drug in formulation (mg)	Amount of drug Pure added (mg)	Total found (mg)	%Recovery of pure drug added
Lopressor (10 mg)	30.24	40.00	69.71	98.68
	30.24	60.00	89.71	99.12
	30.24	90.00	118.31	97.85
Vascoten (10) mg	10.10	20.00	30.23	100.63
	10.10	25.00	35.42	101.28
	10.10	30.00	39.96	99.54

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