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Assessment of long distance chasing photometer (NAG-ADF-300-2) by estimating the drug atenolol with ammonium molybdate via continuous flow injection analysis

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Abstract:

Atenolol was used with ammonium molybdate to prove the efficiency, reliability and repeatability of the long distance chasing photometer (NAG-ADF-300-2) using continuous flow injection analysis. The method is based on reaction between atenolol and ammonium molybdate in an aqueous medium to obtain a dark brown precipitate. Optimum parameters was studied to increase the sensitivity for developed method. A linear range for calibration graph was 0.1-3.5 mmol/L for cell A and 0.3-3.5 mmol/L for cell B, and LOD 133.1680 ng/100 µL and 532.6720 ng/100 µL for cell A and cell B respectively with correlation coefficient (r) 0.9910 for cell A and 0.9901 for cell B, RSD% was lower than 1%, (n=8) for the determination of atenolol at concentration (0.5, 0.7 and 5) mmol/L respectively. The results were compared with classical method UV-Spectrophotometric at λ_{max}=270 nm using the standard addition method via the use of t-test, at 95% confidence level. The comparison of data explain that long distance chasing photometer (NAG-ADF-300-2) is the choice with excellent extended detection and wide application.

Key words: Atenolol, Attenuation of light, Continuous flow injection analysis, Diverged light, Turbidity.

Introduction:

Atenolol chemically known as Benzeneacetamide, 4-[2-hydroxy-3-[(1-methylethyl) amino] proxy] or called Tenormin. White powder, Molecular Formula C_{14} H_{22} N_2O_3, Molecular Weight 266.336 g/mol. Solubility of atenolol in ethanol + Water mixtures (1), solubility in alkaline medium and slightly solubility in water (2). Stability of atenolol in acidic environment depending on diversified polarity, structure of atenolol Fig. 1 (3).

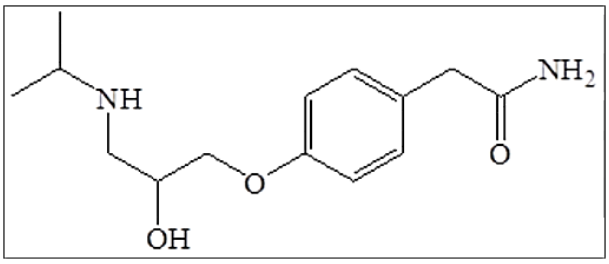

Figure 1. Structure of atenolol

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Atenolol is classified as selective β_1-receptor and is sometimes written β-blockers.
Atenolol drug commonly used for management of hypertension prevention of heart diseases angina pectoris and control of some forms of cardiac arrithymia. The role of atenolol in the treatment of high blood pressure is less favorable than propranolol, as the usual dose carries the risk of diabetes (4,5).Several methods depend on continuous flow injection analysis (6-17) and several analytical methods have been reported for the determination of atenolol such as high performance liquid chromatographic(HPLC) (18-22), Uv-Vis spectrophotometry (4, 23-27), chromatographic densitometry (3), reflectance spectroscopy (28), spectrofluorometry method (29), potentiometric titration (30),GC-Mas (5).

In this work, study and determination of atenolol with ammonium molybdate the obtained resultant signals which resulted from the attenuation of the incident light on particulate surfaces of molybdenum dioxide (i.e.; the precipitated particulate is dark brown in colour of MoO_2). using a new long distance chasing photometer for 300 mm length with 2 mm path length to chase and to accumulate output resulted from attenuated incident

light 0-180^0 via two flow cells of 110 mm and 60 mm length (NAG-ADF-300-2) (31).

Materials and Methods:

Reagents and chemicals

All chemicals were used of analytical-reagent and all the solutions dissolved by distilled water. A standard solution of 50 mmol/L of atenolol, molecular weight 266.336 g/mol, was prepared by dissolving 1.3317 g in 100 ml. A series of ammonium molybdate solutions were prepared from the dilution of standard solution 10 mmol/L with distilled water.

Apparatus

A homemade NAG-ADF-300-2 is a long distance chasing photometer as a flow cell will have 300 mm as a distance with 2 mm as a path length to chaise and to accumulate the output resulted from attenuation of incident light 0-180^0 and diverged or fluorescence light at 0-90^0 via a flow cell. The first flow cell is of 110 mm length covered with 11 white snow LED (WSLED) followed by uncovered distance of 100 mm length then attached to another with 2 solar cell at each side of (0-180^{0}and 0-90^0) cell (cell number 2) which is covered by 6 WSLED and a single photo cell (solar) of 60 mm length at each side was used with peristaltic pump (Ismatec, Switzerland) and six-port medium pressure injection valve (IDEX Corporation, USA) with sample loop (1 mm i.e. Teflon, variable length). Potentiometric recorder to estimate the output signals (Siemens, Germany). UV-Spectrophotometric (RF-1501, shimadzu, Japan) was use for classical methods.

Methodology

Using a manifold of two lines coupled with NAG-ADF-300-2 instrument to determination of atenolol via

It's reaction with ammonium molybdate as shown in fig.2. The first line as a carrier stream at 3.6 ml/min flow rate (distilled water) passing through the injection valve to carry the sample segment of atenolol (5 mmol/L) to meet with the ammonium molybdate (1 mmol/L) in the second line at 5.8 ml/min flow rate at a Y-junction point before it is introduced to the NAG-ADF-300-2 analyser.

The obtained resultant signals which resulted from the attenuation of the incident light on particulate surfaces of molybdenum dioxide (i.e.; the precipitated particulate is dark brown in colour of MoO_2).

It can be noticed that the result obtained from cell A are higher in sensitivity than the cell B output signals.

The higher no. of $WSLED_S$ and solar cell detection available for cell A gives most probably the reason of this higher sensitivity compared with reference to cell B.

Scheme 1. Shows a proposed expected mechanism for the reaction of atenolol with ammonium molybdate in aqueous medium (26, 32).

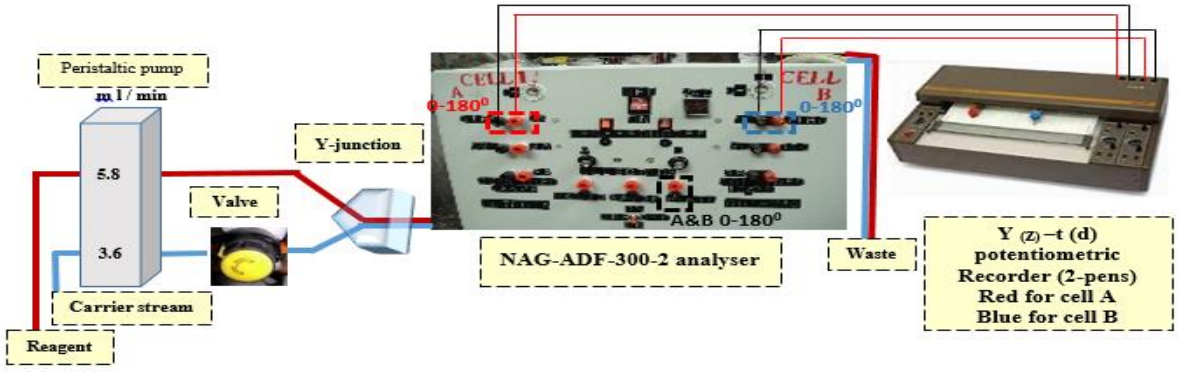
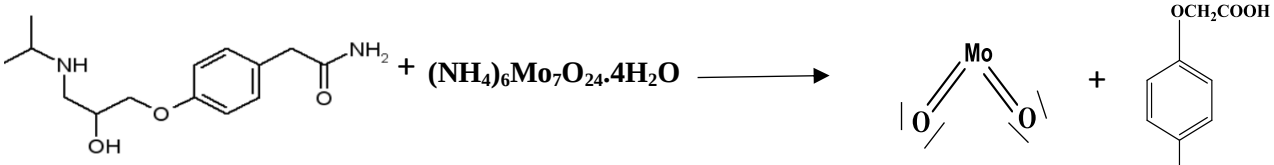


Figure 2. Flowgram manifold system used for the determination of atenolol. Using sample volume 100 µL, [atenolol] = 5 mmol/L, [ammonium molybdate] =1mmol/L, flow rate of carrier stream= 3.6 ml /min, speed of recorder of recorder=60 cm/hr., Intensity I=3 for cell A and I=2 for cell B With Enlarged unit NAG -ADF-300-2 unit (working keys).



Atenolol + **$(NH_4)_6Mo_7O_{24}.4H_2O$** → **Molybdenum dioxide** + **Dark brown Precipitate**

Soluble in OH^- medium

+ NH_{3aq} + $(CH_3)_2CH\,NH\,CH_3$ + H_2O

Scheme 1. Probable mechanism pathway for the oxidation of atenolol by ammonium molybdate

Study of the optimum intensity used for cell A and cell B

In choosing the optimum intensity of used WSLED for either cells (cell no.1-cell A (11WSLED) and cell no.2-cell B (6 WSLED) with in between distance of 100 mm).An arbitrary selected intensity was put into work (an intensity of indication approximate of selector switch for cell A was on no.3. while it was on no.2 for cell B which was based on preliminary experiment).These numbers reflect the 0-1-2-3-4 intensities which were varied a according to nature and type of reaction carried out. On the basis of obtained responses profile (Fig.2) other necessary chemical and physical parameters were carried out, which describe the full detailed study supported by the recorded of $Y_{Z\,(mV)} - t_{\,min}\,(d)_{\,cm}$ response .Figure 3. A and B shows the intensity (I) of the response for either cells from I=1 to I=4.The optimum intensity of the measuring cell A, I=3 and I=2 for cell B which was adopted in subsequent studies.

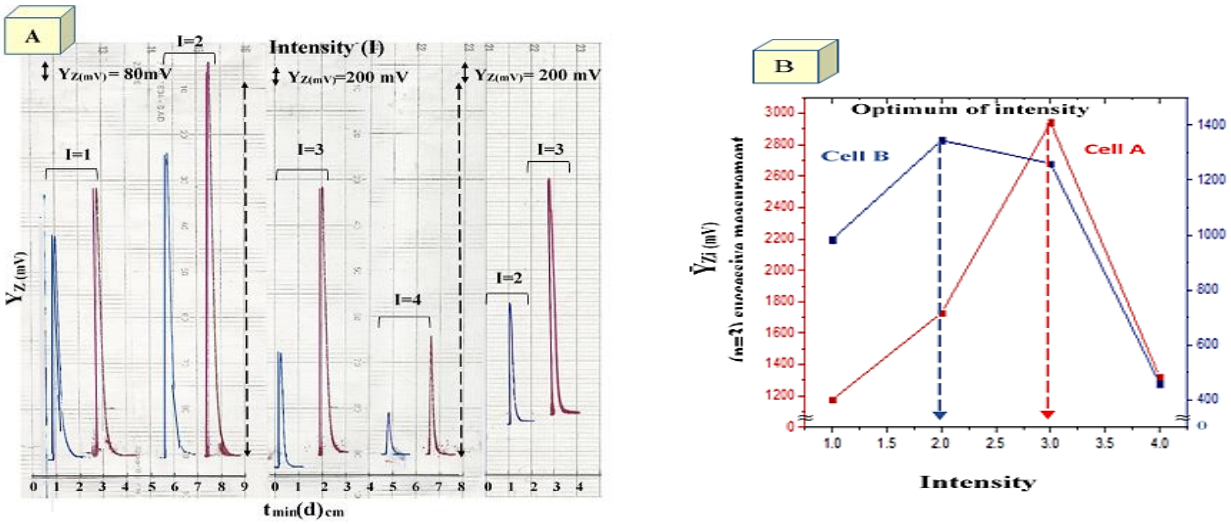


Figure 3. Effect of intensity (I) for either cells (cell A (11 WSLED) and cell B (6 WSLED)) on: A: $Y_{Z\,(mV)} - t_{\,min}\,(d)_{\,cm}$ transducer output response. B: Attenuation of incident light expressed as an average peak heights. Using: Atenolol (5 mmol/L)-Ammonium molybdate (5 mmol/L) system, sample volume 100 µL, 3.2 mL / min flow rate of carrier stream and speed of recorder 60 cm/hr. (1 cm/min).

Results and Discussion:

Optimization of variable

Chemicals parameters (mainly concentration of reagent and type of carrier stream for the atenolol with ammonium molybdate system) as well as physical parameters: sample volume, flow rate were studied using two lines manifold system (Fig.2).

- Chemical variables

- Ammonium molybdate concentration

Variables concentration of precipitating of Ammonium molybdate 0.5-7 mmol/L were prepared of 100 µl sample volume was injected through the carrier stream (distilled water). 5 mmol/L concentration of atenolol was injected with 3.2, 4.6 mL/min flow rate for carrier stream and reagent respectively. In addition to I=3 for cell A, I=2 for cell B. It was found that an increase in peak height expressed as an attenuation of incident light with increase of ammonium molybdate concentration.

It is possible that might be attributed to the increase of coloured precipitate particulate which in turn work on attenuation of part of the incident irradiation light plus it's absorption due to its coloration. In addition to the decrease in the resultant intensity due to its penetration to the precipitate particulate suffering many internal reflection and refraction which in turn to attenuate the incoming penetrating light toward the detection area. While at higher concentration (i. e > 1 mmol/L) might lead to increase agglomeration of precipitated particulate and an increase of inter spatial distances which help to increase penetration light toward the detector and a decrease in the response height. Therefore 1mmol/L was selected as optimum concentration for either cells. The results were summarized in Table 1.

Table 1. Effect of variable concentration of ammonium molybdate on energy transducer response (S/N) of atenolol (5 mmol/L) - ammonium molybdate system, using flow rate for carrier stream and Reagent 3.2, 4.6 mL/min respectively, I=3 cell A, I=2 cell B and 100 µl of sample volume.

[ammonium molybdate] mmol/L	Attenuation of incident light expressed as an average peak heights(n=3) $\bar{Y}_{Z\ i(mV)}$	RSD%	Confidence interval at (95%) $\bar{Y}_{Z\ i(mV)} \pm t_{0.05/2,n-1}\ \sigma_{n-1}/\sqrt{n}$
	Cell A		
0.5	1840	0.0717	1840 ± 3.2792
1	2800	0.0400	2800 ± 2.7824
3	2300	0.0643	2300 ± 3.6767
5	2620	0.0469	2620 ± 3.0556
7	2500	0.0768	2500 ± 4.7698
	Cell B		
0.5	1080	0.1241	1080 ± 3.3289
1	1460	0.0877	1460 ± 3.1799
3	1320	0.0848	1320 ± 2.7824
5	1340	0.0761	1340 ± 2.5340
7	1340	0.1037	1340.4531

$t_{0.05/2,2}$=4.303, $\bar{Y}_{Z\ i\ (mV)}$: (S/N) energy transducer response of cell A and cell B in mV for n=3

-Effect of different salt

The reaction of atenolol (5mmol/L) with ammonium molybdate (1mmol/L) was studied in different media (water, ammonium acetate, potassium nitrate and sodium nitrate) at 10 mmol/L concentration in addition to aqueous medium as a carrier stream. It was noticed for Fig.4. A, B that the studied; salt causes to a decrease of S/N-response; this might be attributed leads to its effect in an increasing the agglomeration i.e.; increase the density of aggregates and compactness with each other than increase the intensity of transmitted light as there will be more vacant spaces in between agglomerates of particulate. On this basis; it 'were (the salts) cancelled throughout this work and distilled water as a carrier stream in the next studied.

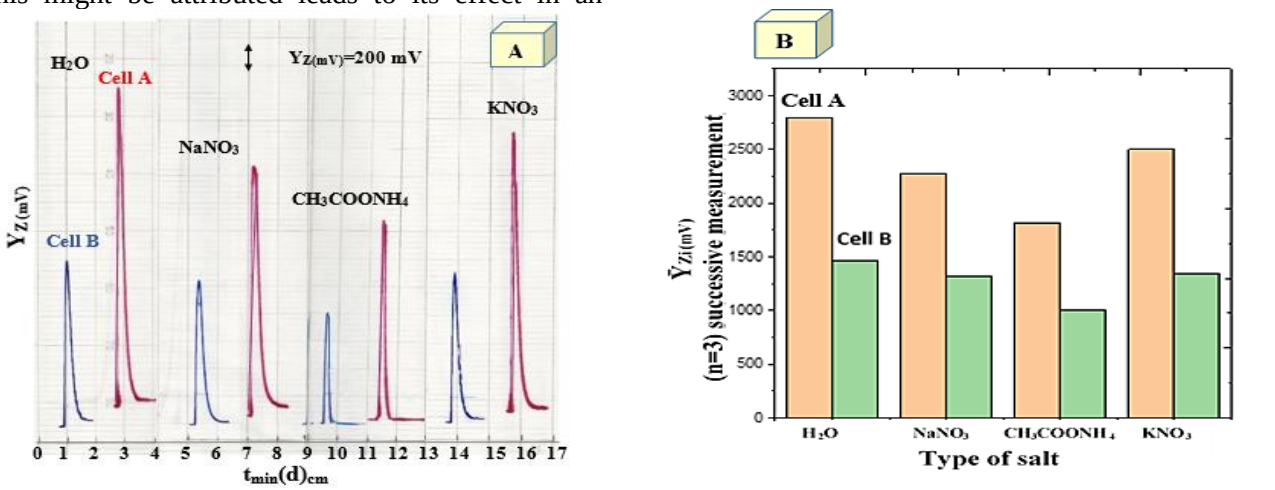


Figure 4. Effect of the different salts on A: S/N energy transducer response versus time using 100 µl Sample volume, flow rate 3.2, 4.6 ml/min of carrier stream and reagent respectively, Atenolol (5mmol/L) - Ammonium molybdate (1 mmol/L) system. I=3 for cell A and I=2 for cell B. B: Attenuation of incident light expressed as an average peak heights.

-Effect of variable concentration of sodium hydroxide

Variable concentration of sodium hydroxide at ranging 2-50 mmol/L were used to dilute the prepared solutions via the use of atenolol (5mmol/L) – ammonium molybdate (1 mmol/L) system , 100 µl sample volume at 3.2 ml/min and 4.6 ml/min flow rate for carrier stream and reagent solution respectively. It was noticed that an increase of NaOH solution causes a decrease in S/N-transducer response, most probably due to friability (flakiness) of precipitated particulate; then more room will be available in not obstructing or attenuated light source effect; or the prevention of the oxidation process for atenolol drug with an output of not forming MoO_2 precipitate. Therefore dilution of the prepared drug solution will be done by distilled water not else. The set of data were summarized in Table 2.

Table 2. Effect of [NaOH] on the measurement of energy transducer response via attenuation of incident Light for determination of atenolol (5 mmol/L) using atenolol –ammonium molybdate (1 mmol/L) System, 100 µl sample volume at 3.2 ml/min and 4.6 ml/min flow rate for carrier stream and Reagent solution respectively, I=3 for cell A and I=2 for cell B.

[NaOH] mmol/L	Attenuation of incident light expressed as an average peak heights (n=3) $\bar{Y}_{Z\ i(mV)}$	RSD%	Confidence interval at (95%) $\bar{Y}_{Z\ i(mV)} \pm t_{0.05/2,n-1}\ \sigma_{n-1}/\sqrt{n}$
	Cell A		
0	2800	0.0400	2800±2.7824
2	1200	0.1100	1200 ±3.2792
5	180	0.8444	180 ±3.7761
10	160	1.2437	160 ±4.9437
50	0*	0*	0*
	Cell B		
0	1460	0.0877	1460±3.1799
2	700	0.2171	700±3.7761
5	120	1.5167	120 ±4.5214
10	80	2.4875	80 ±4.9437
50	0*	0*	0*

0*: Dose not obtained any (S/N) energy transducer response at 50 mmol/L concentration of NaOH $t_{0.05/2,2}$=4.303, $\bar{Y}_{Z\ i\ (mV)}$: (S/N) energy transducer response of cell A and cell B in mV for n=3

-Physical variables

-Flow rate

Flow rate study was carried out using the unit cell of two lines as shown in Fig.45.A to assess the $Y_{Z\ (mV)}$-$t_{\ min}$ (d) $_{cm}$ response profile. It was noticed (Fig. 5. A). that an increase in S/N- response profile from cell A & cell B up to 3.6 ml/min for carrier stream. This may be attributed to an increased opportunity for the crystal that are formed to grow up relatively; while there is a time lag difference between cells used.

At high speed (i.e.; more than 3.6 ml/min (carrier stream)) does not offer an a time lag period causing increase attenuation of incident light that is measured by the detector. So at high speed, it is noticed that a decrease in response height might be attributed to incomplete or immature precipitation. Therefore; 3.6 & 5.8 ml/min flow rate for carrier stream and reagent respectively for either cells (Fig.5.B and C).

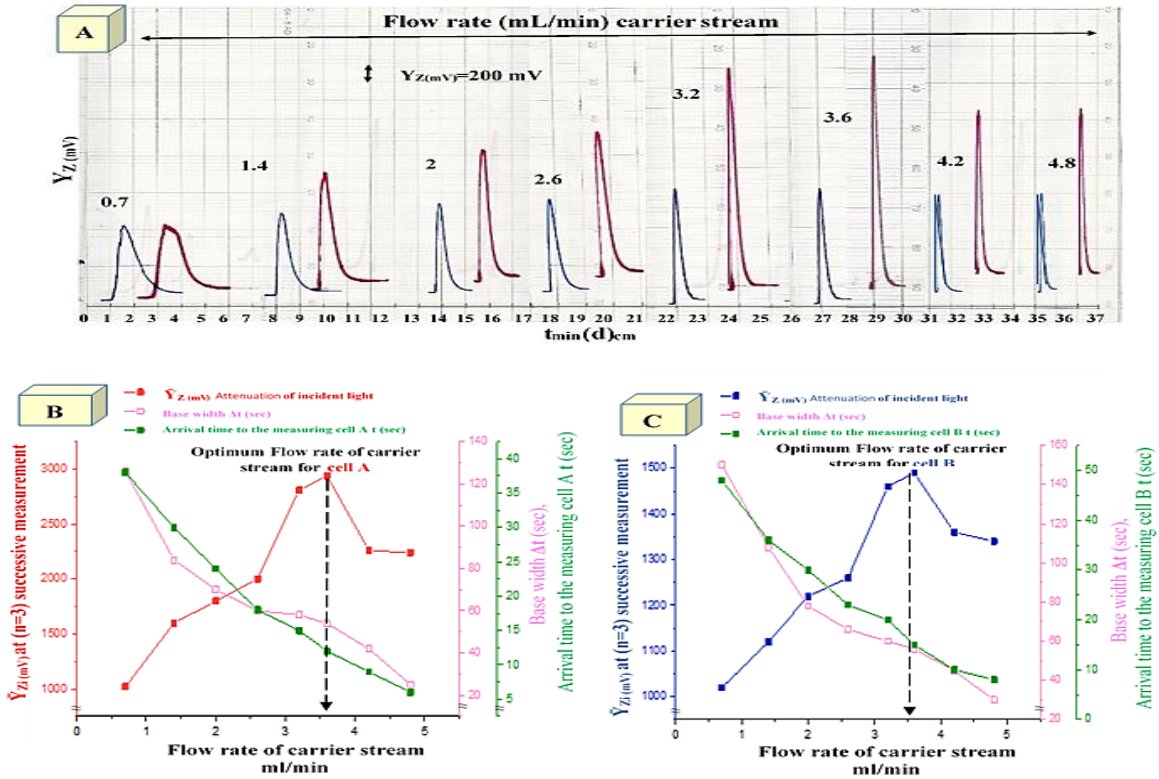


Figure 5. Effect of the variation of flow rate on: A: S/N energy transducer response versus $t_{\ min}$ (d) $_{cm}$ B, C: Attenuation of incident light expressed as an average peak heights in mV ($\bar{Y}_{Z\ i\ (mV)}$) for cell A, cell B using atenolol (5 mmol/L) - ammonium molybdate (1 mmol/L) system, 100 µL of sampleVolume, the intensity of light expressed as I=3 for cell A and I=2 for cell B.

-Sample volume

A sample volume will represent the analyte concentration to be determined. Variable length of Teflon tube ranging (2.6-25.5) cm of diameter (D) 1 mm that is equivalent to (20-200) µL of sample volume. Therefore and on this basis a clear response profile is required for the chosen reaction intended to be carried out.

Large volume is not a recommended methodology in CFIA. As this kind of volume will form by local concentration for the precipitate that can dissolve i.e.; at least part of it due to the continuous flow of carrier stream that will cause the k.s.p of the precipitate > than the multiplication of the concentration of reacted species raised to power of its sharing strength of contribution.

Low sample volume will represent a quick transit of reaction product in front the detector (solar cells which are not of fast response detection compared to PMT that can detected at Nano sec. level).Therefore a very small sample segment might be not a convenient level of sample volume. A compromise was made in this study to choose 100 µL as a suitable most convenient of sample size level.

By this broadening as well as disturbed $Y_{Z(mV)} - t_{min}$ $(d)_{cm}$ response were avoided to obtain trustable more confident; a way of interference caused by precipitated aggregate that might cause a delayed movement of reacting product. The obtained results tabulated in Table 3.

Table 3. Effect of the variation of sample volume on the Attenuation of incident light by 5 mmol / L Concentration of atenolol and 1 mmol / L concentration of ammonium molybdate, speed of Recorder 60 cm/hr. and flow rate of the carrier stream 3.6 ml/min.

Length of Sample segment Cm r=0.5m	Sample volume µL $V=\pi r^2h$	Attenuation of incident light expressed as an average peak heights (n=3) $\bar{Y}_{Z\ i(mV)}$	RSD%	Confidence interval at (95%) $\bar{Y}_{Z\ i(mV)} \pm t_{0.05/2,n-1}\ \sigma_{n-1}/\sqrt{n}$	t (sec)	Base width Δ t (sec)	V_{add} (ml) at flow cell	Concentration mmol/L at flow cell	D f at flow cell
				Cell A					
2.60	20	1940	0.0784	1940±3.7761	6.0	36	5.6600	0.0177	283.00
3.10	24	2160	0.0856	2160±4.5959	7.2	39	6.1340	0.0196	255.58
4.10	32	2200	0.0764	2200±4.1736	8.4	42	6.6120	0.0242	206.61
7.00	55	2180	0.0789	2180±4.2729	9.6	48	7.5750	0.0363	137.74
9.00	71	2300	0.0857	2300±4.8940	10.8	51	8.0610	0.0440	113.64
12.74	100	2940	0.0415	2940±3.0308	12.0	54	8.5600	0.0584	85.62
20.40	160	2240	0.0915	2240±5.0927	15.0	72	11.4400	0.0699	71.53
25.50	200	2260	0.1137	2260±6.3846	18.0	78	12.4200	0.0805	62.11
				Cell B					
2.60	20	1140	0.1728	1140±4.8940	6.8	40	6.2867	0.0159	314.47
3.10	24	1340	0.1381	1340±4.5959	8.0	45	7.0740	0.0170	294.12
4.10	32	1260	0.1571	1260±4.9188	9.0	47	7.3953	0.0216	231.48
7.00	55	1300	0.1192	1300±3.8506	10.6	50	7.8883	0.0349	143.27
9.00	71	1380	0.1435	1380±4.9188	13.0	54	8.5310	0.0416	120.19
12.74	100	1490	0.0826	1490±3.0556	15.0	56	8.8733	0.0563	88.81
20.40	160	1400	0.1421	1400±4.9437	18.0	75	11.9100	0.0672	74.41
25.50	200	1400	0.1800	1400±6.2604	20.0	80	12.7333	0.0785	63.69

t: Arrival time from injection valve reaching to measuring cell (sec) ,Δ t: Base width of peak(sec), $t_{0.05/2,2}$ =4.303,Df : Dilution factor at flow cell

-Effect of reaction loop length

Effect of including a coil (reaction, delay, exchange, adsorption, or reduction and oxidation (i. e change of valence of selected reaction ions)) in the flowgram in CFIA used throughout this research work; as part of the designed unit for determination of atenolol. The reaction coil of any kind whether it is made of glass or Teflon as turns (depending on length, loop diameter, and tube diameter used).Glass is preferred material due to its ability that it can be cleaned easily via pumping a certain cleaning solvent to remove precipitated particulate (usually at high concentration of reactants or even sedimentation of precipitate particulate due to high molecular weight of precipitated reaction product or precipitated reagent that is used (e.g. change of valence). Usually any addition of extra length to the manifold will increase dispersion via different process (e.g. convection at high speed of flow rate or diffusion at low speed of flow rate).

On this basis reaction coils are avoided unless it serve the purpose of using or verifying its use. The volume of the reaction coil can be found as it is a cylinder of cross section diameter of ϕ =2r (where r

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= radius; i.e. tube radius). A volume of a cylinder $=\pi r^2 L$ (where L= length of the used tube for e. g is ϕ=1, the r=0.5 mm for 100 mm length. The volume will be equal to 3.14(0.05 cm) ^{2} × 20 cm=0.157 cm^3=157 µL.

So, it was found that using different length of reaction coil (variable volume) will be introduced leading to dilution of sample segment reaction product which that ladies' to an even distribution when used of a very high volume, will lead to a highly dispersed of precipitate which cause a weaker signal (or even undetected signal). Therefore a compromise of using a convenient reaction coil length (fig. 6. A,B and C) and in watching the results of measurement shows that a direct measurement with no coils in the manifold system will the way of the system works i.e., better with very good excellent output, in addition to trust ability of S/N response and $Y_{Z(mV)} - t_{min}$ $(d)_{cm}$ response profile. Figure 6. A shows a kind of response.

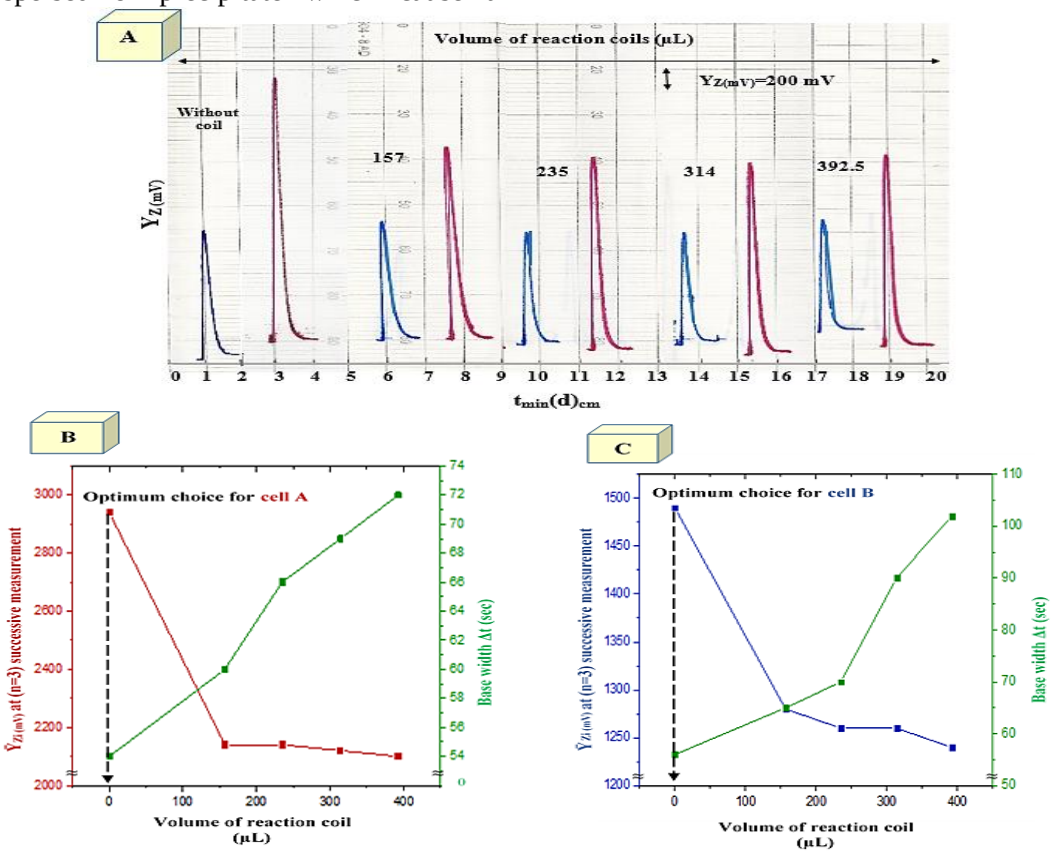

Figure 6. Effect of reaction coil on: A: S/N- response of the energy transducer versus t_{min} $(d)_{cm}$ B, C: Attenuation of incident light expressed as an average peak heights in mV ($\bar{Y}_{Z\,i\,(mV)}$) for cell A and cell B respectively by atenolol (5 mmol/L) - ammonium molybdate (1 mmol/L) system at Flow rate of the carrier stream 3.6 ml/min, I=3 for cell A and I=2 for cell B.

- Study of the optimum intensity used for While Snow Light Emitting Diodes (WSLEDS) in NAG-ADF-300-2 analyser

A study was conducted for the effect of intensity of incident light for the irradiation sources on the S/N – response of the energy transducer via the selector switch (C.F front panel diagram of NAG-ADF-300-2.The selector switch gives 0-1-2-3-4 i.e.; four choices plus the off position for both cell individually controlled.

It was noticed from a selection of 3 position (i.e.; I=3) was very convenient intensity for cell no.1 (cell A) (larger number of the selector switch means more light intensity).while position 2 (I=2) of the selector switch was a convenient intensity. It was same intensity chosen in first of atenolol study of assessment. The higher intensity (I=3) for cell A refers to that precipitated particulate that is formed are line particles and dense; therefore high light intensity is required. While it, was not necessary to use high light intensity for cell B due to conglomeration of precipitated particulates and inter spaces are formed. Therefore, a low intensity of height is required for cell B. The results summed up in table 4.

So, it was concluded that the intensity that was chosen at the beginning (at preliminary experiments) remained as it was.

Table 4. Effect of intensity on attenuation of incident light expressed as an average peak heights (mV) For determination of atenolol (5 mmol/L) - ammonium molybdate (1 mmol/L) system, speed of Recorder 60 cm/hr., flow rate of the carrier stream 3.6 ml/min and sample volume 100 µL.

Intensity(I)	Attenuation of incident light expressed as an average peak heights (n=3)$\bar{Y}_{Z\ i(mV)}$	RSD%	Confidence interval at (95%) $\bar{Y}_{Z\ i(mV)} \pm t_{0.05/2,n-1}\ \sigma_{n-1}/\sqrt{n}$
		Cell A	
1	1000	0.1940	1000±4.8195
2	1420	0.0789	1420±2.7824
3	2400	0.0638	2400±3.8009
4	1820	0.0780	1820±3.5277
		Cell B	
1	920	0.1989	920±4.5462
2	1220	0.1246	1220±3.7761
3	1200	0.1650	1200±4.9188
4	440	0.2818	440±3.0805
	Cell A & Cell B	Cell A & Cell B	Cell A & Cell B
Cell A(I=2) & Cell B (I=3)	1300 &1160	0.1169 & 0.1276	1300±3.7761 & 1160±3.6767
Cell A(I=3) & Cell B (I=2)	2940 & 1490	0.0415 & 0.0826	2940±3.0308 & 1490±3.0556

The intensity of output response is varied for cell A and cell B

-Estimating the linear dynamic range from scatter plot for the variation of atenolol versus S/N Energy transducer response

Using the optimum chemical and physical parameters; a series of atenolol solutions (0.1-20 mmol/L) were prepared this will represent the x-axis (Independent variable).

The attenuation of incident light that was measured gave the following S/N energy transducer responses as Y here represent the dependent variable as shown in Fig.7; in which, the height of response increased when the analyte of concentration is increased. The directly proportional up to 9 mmol/L and 7 mmol/L for cell A and cell B respectively (Fig.8.A, B) between variation precipitate particulate formation and concentration might be attributed to increase of many such as: internal reflection, refraction, absorbance and diverged light from within the precipitated particles when the beam of light diffused inside of particles. In addition to abstraction of light by the precipitated particulate; while gives at the end for all these factors combined all together, the measurement that is due to was only at 0-180^0 angle. Fig. 8. A, B explains the variance ranges for each cells. (i.e.; scatter plot at range (0.1-20) mmol/L, dynamic range (0.1-9) mmol/L, working range (0.1-7) mmol/L and linear dynamic range (0.1-3.5) mmol/L for cell A and scatter plot at range (0.1-20) mmol/L, dynamic range (0.1-7) mmol/L, working range (0.2-5) mmol/L and linear dynamic range (0.3-3.5) mmol/L for cell B).

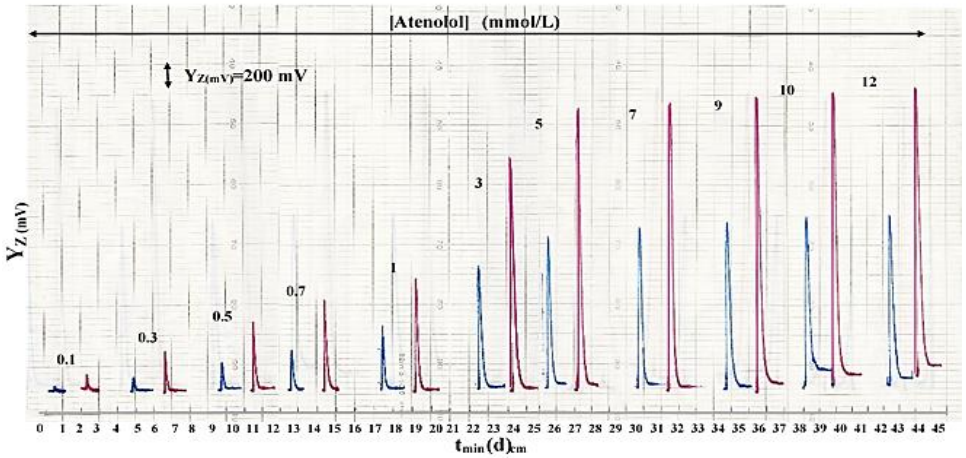

Figure 7. Some of response profile versus time using ammonium molybdate (1 mmol/L), sample Volume 100 µL.I=3 for cell A and I=2 for cell B.

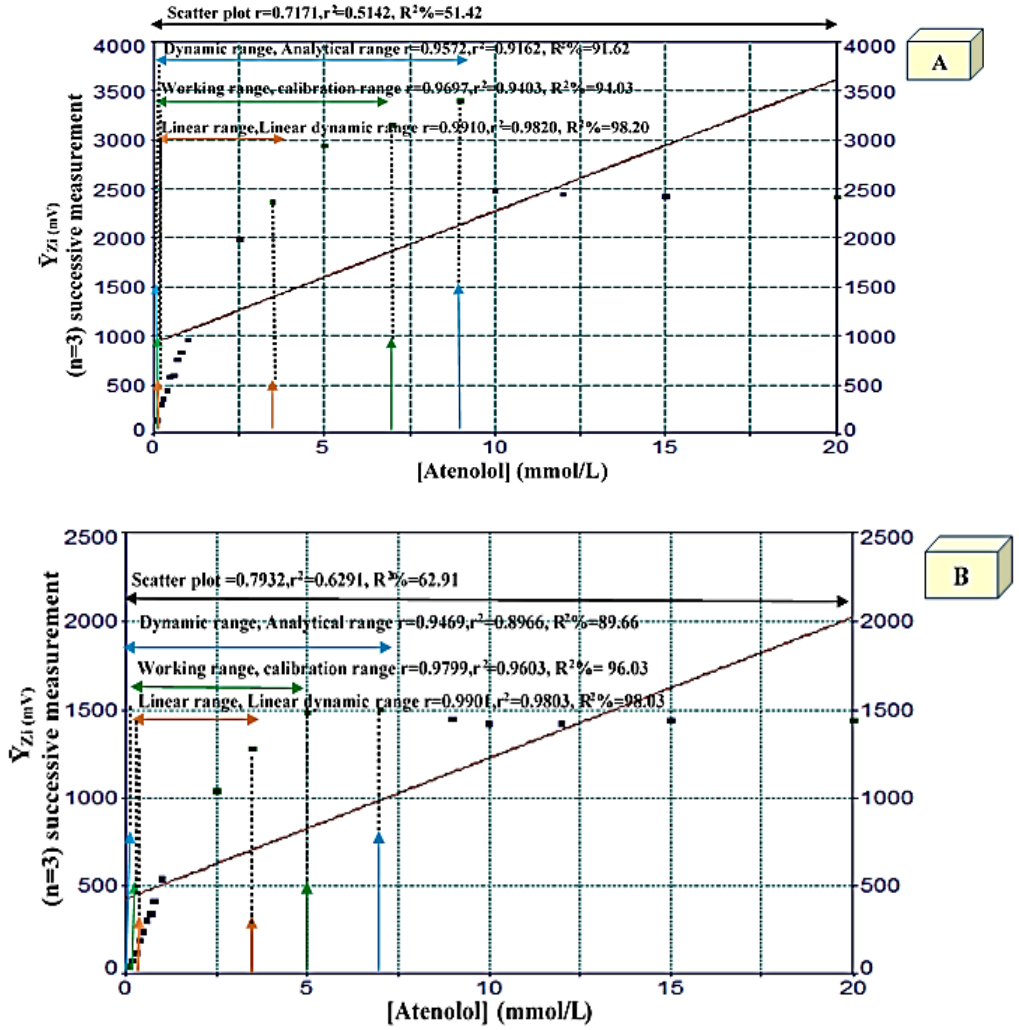

Figure 8. Different range for the effect of atenolol concentration on attenuation of incident light using NAG-ADF-300-2, A): for cell A, B): for cell B

While increasing the concentration more than 9 and 7 mmol/L for cell A and cell B respectively causing that the signal (S/N) energy transducer independent on concentration, It is might be attributed to agglomeration of particulate and increase of inter spatial distances which lead to increase of transmitted light toward the detector.

-Limit of Detection

In general terms, the L.O.D of an analyte may be described as that: concentration which gives an instrument signal y significantly different from the blank or back ground signal. This description gives the analyst a good deal of freedom to decide the exact definition of L.O.D. There is an increasing trend to define the L.O.D. as: the analyte concentration giving a signal equal to the blank signal, y_B plus three standard deviation of the blank S_B.

$$\mathbf{L.O.D = y_B + 3S_B}$$

We have been using three approaches for the expression of L.O.D

1. Gradual dilution. Practically based on successive dilution of the lowest concentration used in calibration graph, this should be regarded as the real, and trustable value of D.L. i. e. Reliable D.L. for the proposed method .

2. Theoretically (slope method)

$$\mathbf{L.O.D. = 3S_B / slope}$$

$S_B = \sigma_{n-1}$ B standard deviation of blank n=13

3. Theoretically (Linear equation) method

$$\mathbf{\hat{Y} = y_B + 3S_B}$$

Y_B: average response for the blank solution, this is equivalent to intercept (a) in straight line equation

$$\mathbf{y=a + b\,x}$$

The last two methods are an output of a linear regression graph treatments where the obtained (real) results are subjected to statistical treatments, these method can be used as an approximate indication but should not unless otherwise defined.

A study was carried out to calculate the limit of detection of atenolol-ammonium molybdate (1 mmol/L) system through three methods as tabulated in Table 5.

Table 5. Limit of detection for atenolol at optimum parameters using 100µl as an injection Sample, 3.6 ml/min flow rate for carrier stream, [ammonium molybdate] 1mmol/L

Type of cell	Practically based on the gradual dilution for the minimum concentration (0.1 mmol/L)	Theoretical based on the value of slope x=3S_B/slope	Theoretical based on the linear equation $\hat{Y} = Y_b + 3S_b$	n
Cell A	0.005 mmol/L (80 mV) 133.1680 ng/100 µL	36.7144 ng/100 µL	12.1209 µg/100 µL	11
Cell B	0.02 mmol/L (34 mV) 532.6720 ng/100 µL	66.5787 ng/100 µL	13.3328 µg/100 µL	9

X=L.O.D based on slope and S_B= standard deviation of blank repeated for 13 times. : Y_b average response for blank= intercept (a), S_b: standard deviation equal to S y/x (residual): $\hat{Y}$ estimated response (mV), n: number of injection

-Repeatability

The relative standard deviation expressed as percentage which is equally to the repeatability of the measurement. A repeated measurements for eight successive injections were measured at fixed concentrations of atenolol for three concentrations were used 0.5, 0.7, 5 mmol/L respectively in optimum parameters. The obtained results is tabulated in Table 6 is shown of repeatability at 0.5, 0.7, 5 mmol/L respectively. In addition to study of repeatability with minimum of the RSD% which equal to 1%.

Table 6. Repeatability of atenolol at optimum parameters with 100 µl sample volume

[Atenolol] mmol/L	Attenuation of incident light expressed as an average peak heights (n=8) $\bar{Y}_{Z\,i(mV)}$	RSD%	Confidence interval at (95%) $\bar{Y}_{Z\,i\,(mV)} \pm t_{0.05/2,n-1}\,\sigma_{n-1}/\sqrt{n}$
	Cell A		
0.5	580	0.2276	580±1.1037
0.7	760	0.2092	760±1.3295
5	2940	0.0752	2940±1.8479
	Cell B		
0.5	240	0.7417	240± 1.4884
0.7	340	0.4088	340±1.1623
5	1490	0.1067	1490±1.3295

n=8 number of injection,$t_{0.05/2,7}$=2.365

-Classical method of UV- Spectrophotometric

The assessment evaluation of the new developed methodology (i.e.; NAG-ADF-300-2 analyser) for the determination of atenolol using atenolol - ammonium molybdate (1 mmol/L) system was compared with the available literature method, namely UV-Spectrophotometric method (33) which was based on the measurements of absorbance for the range of concentration 0.01-6 mmol/L at λ_{max}= 270 nm using quartz cell. Table 7 shows the variable data treatments. The detection limit was 0.005 mmol/L (5 µmol/L) equivalent to 1.3317 µg / sample.

Table 7. Different ranges for the atenolol concentration versus absorbance using spectrophotometer (Classical method)

Type of mode	Range of [atenolol] mmol/L(n)	$\hat{Y}_{Z\,i} = a \pm S_a t + b(\Delta y / \Delta x_{mmol/L}) \pm S_b t$ [Atenolol] mmol/L at confidence level 95%,n-2	r, r^2, R^2%	t_{tab} at 95%,n-2	Calculated t-value $t_{Cal} = r/\sqrt{n-2} / \sqrt{1-r^2}$
Scatter plot	0.01-6 (18)	0.3291 ±0.2033+0.3706±0.0856 [Atenolol] mmol/L	0.9167,0.8404,84.04	2.120 < 9.1785	
Dynamic range or analytical range	0.01-5 (17)	0.2632±0.1750+0.4449±0.0891 [Atenolol] mmol/L	0.9396,0.8829,88.29	2.131 < 10.6344	
Working range or calibration range	0.01-4 (16)	0.1926±0.1386+0.5371 ± 0.0871 [Atenolol] mmol/L	0.9623,0.9260,92.60	2.145 << 13.2375	
Linear range or linear dynamic range	0.05-2.5 (13)	0.0864±0.0599+0.7157± 0.0548 [Atenolol] mmol/L	0.9934,0.9869,98.69	2.20128.7747	

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-Assessment of NAG - ADF- 300 - 2 analyser using two cell and multi solar cells for the Determination of atenolol in drugs

The newly developed methodology (NAG-ADF-300-2) was used for the determination of atenolol in three different samples of drugs from three different of companies (Atenolol, Bristol, UK, 100 mg),(Vascoten, medochemie, Cyprus, 100 mg) and (Nova ten, Ajanta, India, 100 mg).

The continuous flow injection analysis used of homemade NAG-ADF-300-2 which that mean a long distance chasing photometer for 300 mm length with 2mm path length to chase and accumulate output response from attenuation of incident light at $0\text{-}180^0$ via the use of two cells of 110 mm (cell A) and 60 mm length (cell B) and was compared with UV-spectrophotometric method via the measurement at λ_{max}=270 nm.

A series of solution were prepared of each drug (20 mmol/L) by transferring of 0.5 mL to each of the five volumetric flask (10 mL) followed by the addition of 0.0, 0.2, 0.4, 0.6, 0.8 mL from 50 mmol/L of standard solution to obtain 0,1,2,3,4 mmol/L for developed method ,while classical method (20 mmol/L) by transferring of 0.5 mL to each of the five volumetric flask (10 mL) followed by the addition of 0.0, 0.04, 0.06, 0.08, 0.1 mL from 50 mmol/L of standard solution of atenolol to obtain 0,0.2,0.3,0.4,0.5 mmol/L. Taking into a consideration that the first flask is for the sample. The measurements were conducted by both methods. Results were mathematically treated for the standard addition method. Table 8. A and B were shown a practical content of active ingredient at 95% confidence level & efficiency of determination in addition to paired t-test which shows a comparison at two difference paths (34,35).

First test: Comparison of Newly developed method (NAG-ADF-300-2) analyser with official quoted value B.P (36) (100 mg) as shown in table 8. B (column 5) by calculated t-values of each individual company and this comparison with tabulated t-value.

A hypothesis can be estimated as follow

Null hypothesis: There is no significant difference between the means obtained from three source of three different companies ($\bar{w}_i$) and quoted value (μ)

i.e.; $H_o: \bar{w}_i = \mu$

For: Atenolol (Bristol, 100 mg, UK), Vascoten (Medochemie, 100 mg, Cyprus) and Nova ten (Ajanta, 100 mg, India) companies.

Against:

Alternative hypothesis: there is a significant difference between the means and quoted value

i.e.; $\bar{w}_o \neq \mu$ for: three different companies

Since some value obtained $t_{cal} > t_{tab}$ (4.303) at confidence level 95% and degree of freedom =2; Null hypothesis will be reject and accepting the alternative hypothesis; these mean that there is a significant difference between the quoted active ingredient value and the measured value. One this base; the newly developed method can be used equally well as standard reference methods. Another obtained t_{cal} –value indicate that there was no significant different between the newly developed method and claimed method by the company as calculated t – value is less than tabulated t – value.

So, the newly method can be used as an alternative analysis method for the determination of atenolol in different drugs.

Second test: Using paired t – test at $\alpha = 0.05$ (2-tailed) for the comparison of developed method using NAG-ADF-300-2 analyser and classical method using shimadzu (UV-1800 double beam) spectrophotometer as shown in Table 8. B (column 6).Taking into the consideration that all drugs from different companies are the same population i.e.; neglecting individual differences between one manufacturer and another.

Assumption

Null hypothesis H_o: $\mu_{\text{NAG-ADF-300-2 analyser}} = \mu_{\text{UV-SP.}}$ There is no significant difference between the mean of different two methods.

An alternative hypothesis: There is a significant difference between the mean of classical method and NAG-ADF-300-2 analyser

i.e.; Alternative H_1: $\mu_{\text{NAG-ADF-300-2 analyser}} \neq \mu_{\text{UV-SP.}}$ The obtained results indicate clearly that there was no significant differences between newly developed method and UV- spectrophotometric (classical method) at 95% ($\alpha = 0.05$) confidence level as the calculated t_{cal} (3.996 and 0.4053) is less than t_{tab} (4.303) for each cell (i.e.; cell A & cell B) for the determination of atenolol in pharmaceutical drugs as shown in Table 8. B (column 6).

Table 8. A: Standard addition results for the determination of atenolol in three pharmaceutical preparation using NAG-ADF-300-2 analyser for cell A, cell B and classical methods.

No. of sample	Commercial name ,CompanyContent Country	Confidence interval for the average Weight of Tablet $\bar{w}$ i ± 1.96σ_{n-1}/√n at 95% (g)	Weight of sample equivalent to 1.33168 g(20 mmol/L)of the active ingredient Wi (g)	Theoretical content for the active ingredient at 95% (mg) Wi ±1.96σ_{n-1} /√n	Type of method: Newly developed methodology Cell A Cell B — UV- Sp. Classical method (Absorbance measurement at λ_{max}=270 nm) — Atenolol mmol/L					Equation of standard addition at 95% for n-2	r , r^2 , R^2%
					0	0.20 ml	0.40 ml	0.60 ml	0.80ml	$\hat{Y}_{Z\,i\,(mV)}=a_{m\,V}\pm S_a t+ b\,(\Delta\, y_{\,mV}\,\Delta\, x_{\,mmol/})\pm S_{\,b}\, t$[Atenolol]mmol/L	
					0	1.00	2.00	3.00	4.00		
					0	0.04 ml	0.06 ml	0.08 ml	0.10ml	$\hat{Y}_{Z\,i}=a\pm S_a t+ b\,(\Delta\, y / \Delta x_{mmol/L})\pm S_{\,b}\, t$[Atenolol]mmol/L	
					0	0.20	0.30	0.40	0.50		
1	Atenolol Bristol 100 mg UK	0.4190±0.00204	5.5801	100±0.4868	490	940	1400	1810	2390	472±117.6048+467±48.0119 [Atenolol]mmol/L	0.9984,0.9969,99.69
					270	600	950	1200	1500	292±77.4216+306±31.6071 [Atenolol]mmol/L	0.9984,0.9968,99.68
					0.392	0.511	0.518	0.551	0.612	0.4024±0.0528+0.4086±0.1604[Atenolol] mmol/L	0.9780,0.9564,95.64
2	Vascoten Medochemie 100 mg Cyprus	0.4030±0.0015	5.3661	100±0.3723	500	1088	1560	2078	2500	547.2000±120.7719+499±49.3048 [Atenolol]mmol/L	0.9985,0.9971,99.71
					330	530	900	1165	1`450	300±98.9752+287.5000±40.4063 [Atenolol] mmol/L	0.9971,0.9942,99.42
					0.592	0.681	0.751	0.812	0.856	0.5860±0.0274+0.5442±0.0834 [Atenolol]mmol/L	0.9965,0.9931,99.31
3	Novaten Ajanta 100 mg India	0.4028±0.0031	5.3643	100±0.7696	389	790	1220	1580	2000	393.400±43.1253+401.200±17.6057[Atenolol]mmol/L	0.9997,0.9994,99.94
					270	580	870	1180	1360	296±108.7480+278±44.3962 [Atenolol] mmol/L	0.9962,0.9925,99.25
					0.351	0.423	0.459	0.511	0.532	0.3504±0.0204+0.3745±0.0620 [Atenolol]mmol/L	0.9960,0.9920,99.20

Ŷ: Estimated response in mV for developed method and absorbance for UV-S p. method, r: correlation coefficient, r^2: coefficient of determination, R^2% (percentage capital R squared): explained variation as a percentage total variation, UV– Sp.: UV –Spectrophotometric method, t0.05/2, □=1.96 at 95%,t0.05/2,3=3.182 for n=5.

Table 8. B: Summary of results for practical content, efficiency (Rec %) for determination of atenolol in three sample of pharmaceutical preparation and paired t-test using two methods.

	Type of method					
	Newly developed methodology Cell A Cell B UV-Sp. Classical method Absorbance measurement at λ_{max}=270 nm					
No. of sample	Practical concentration (mmol/L) in 10 ml in 250 ml	Practical weight of atenolol $\bar{W}$ i (g) ±4.303 $\sigma_{n-1}/\sqrt{n}$ Weight of atenolol in tablet $\bar{W}$ i (mg) ±4.303 $\sigma_{n-1}/\sqrt{n}$	Efficiency of determination Rec.%	Individual t-test for compared between quoted value &practical value $(\bar{W}$ i -μ)$\sqrt{n}$ /σ_{n-1}	Paired t –test Compared between two methods $t_{Cal} = \bar{X}$ d $\sqrt{n}/\sigma_{n-1}$	t_{tab} at 95% confidence level(n-1)
1	1.0107 20.2140	1.3459 ±0.0250 101.0695 ±1.8774	101.0695%	2.4513 < 4.303		
	0.9542 19.0848	1.2707 ±0.0340 95.4233 ±2.5532	95.4233%		cellA	
	0.9848 19.6960	1.3114 ±0.0270 98.4788 ±2.0275	98.4788%	/-7.7134/ > 4.303		
	1.0966 21.9320	1.4603 ±0.0320 109.6594 ±2.4030	109.6594%	17.2961 >> 4.303	$\bar{X}$d=3.0205 σ_{n-1}=1.3092 3.9961< 4.303	
2	1.0435 20.8694	1.3896 ±0.0130 104.3458 ±0.9762	104.3458%			
	1.0768 21.5360	1.4340 ±0.0780 107.6792 ±5.8570	107.6792%	19.1579 >> 4.303	cell B	
	0.9806 19.6110	1.3057 ±0.0670 98.0538 ±5.0315	98.0538%	/-1.6644/ < 4.303		
3	1.0647 21.2948	1.4179 ±0.0490 106.4732 ±3.6795	106.4732%		$\bar{X}$ d=2.1758 σ_{n-1}=9.2970 0.4053< 4.303	
	0.9356 18.7128	1.2460 ±0.083 93.5633 ±6.2325	93.5633%	7.5695 > 4.303		

μ: quoted value, $\bar{w}$: practical content mg, x d: average of difference between two type of method (developed& classical), n (no. of sample) = 3, $\sigma_{n-1:}$ standard deviation, $\bar{w}$ i: practically weight in mg, $t_{0.05/2,2}$=4.303

Conclusion:

The assessment of long distance chasing photometer (NAG-ADF-300-2) through this research work was applied using comparison between NAG-ADF-300-2 analyser with classical UV-Spectrophotometric method. It was recognized that a narrower range is obtained with UV-Spectrophotometric, while a wider range was the characteristic of NAG-ADF-300-2 analyser. A long distance chasing photometer (NAG-ADF-300-2) is the choice with excellent extended detection and a wider applicability. In the future using a new long distance chasing photometer as a flow cell will have 300 mm as a distance with 2 mm as a path length to chaise and to accumulate the output resulted from Attenuation and the Diverged or Fluorescence light at 0-90° via two flow cells of 110 mm and 60 mm length (NAG-ADF-300-2) for study and determination of some selected drugs.

Conflicts of Interest: None.

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تقييم مطياف المسافة التتبعية الطويل من خلال تقدير عقار الاتينولول مع مولبيدات الامونيوم بوساطة تقنية الحقن الجرياني المستمر

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الخلاصة:

تم استخدام عقار الاتينولول مع مولبيدات الامونيوم لاثبات كفاءة وموثوقية وتكرارية مطياف المسافة التتبعية الطويل NAG-ADF-300-2 المحلي الصنع باستخدام تقنية الحقن الجرياني المستمرة. تستند هذه الطريقة على التفاعل بين الاتينولول ومولبيدات الامونيوم في الوسط المائي وتكوين راسب بني غامق من MoO_2. تمت دراسة الظروف المثلى لزيادة حساسية الطريقة المطورة وتم الحصول على مدى خطية لمنحني المعايرة 0.1 -3.5 ملي مول /لتر للخلية الاولى A و0.3-3.5 للخلية الثانية B وحدود كشف LOD 133.1680 نانوغرام/ 100مايكرولتر للخلية الاولى و532.6720 نانوغرام/ 100 مايكرولتر للخلية الثانية ومعامل الارتباط (r) 0.9910 و0.9901 للخلية الاولى والثانية على التوالي والانحراف القياسي النسبي المئوي RSD% اقل من 1% للتراكيز 0.7,0.5 و5 ملي مول / لتر (n=8) للخليتين. تم مقارنة النتائج مع الطريقة الطيفية التقليدية عند الطول الموجي 270 نانوميتر باستخدام طريقة الاضافات القياسية واجراء اختبار t-test للمقارنة بين الطريقتين لتقدير الاتينولول واثبتت النتائج عدم وجود فرق جوهري بين الطريقتين عند مستوى الثقة 95 %. تظهر المقارنة بين االطريقتين ان الطريقة المطورة هي الاختيار الامثل ولمدى كشف ممتاز وتطبيق اوسع.

الكلمات المفتاحية:اتينولول،توهين الضوء،التحليل بالحقن الجرياني المستمر،التعكرية، ضوء متباعد .