UV Spectrophotometric Estimation of Acipimox in Bulk and Capsule Dosage Form

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Abstract: A new simple, rapid, accurate, sensitive and precise spectrophotometric method in ultra violet region has been developed for determination of acipimox (ACX) in bulk and capsule dosage form. Acipimox exhibited maximum absorbance at 231 nm with apparent molar absorptivity of 1.5104×10^4 in distilled water. Beer's law was found to be obeyed in the concentration range 1-10 $\mu\text{g mL}^{-1}$. Correlation coefficient was found to be 0.9998. The developed method was validated respect to linearity, precision, accuracy. The proposed method is useful for the routine estimation of ACX in bulk and capsule dosage form.

Keywords: UV Spectrophotometry, Acipimox, Beer's law, Capsule analysis.

Introduction

Acipimox, chemically 5-methylpyrazine carboxylic acid 4-oxide (Figure 1), is a nicotinic acid analogue which is an antilipolytic drug used in the management of different forms of hyperlipidaemia¹⁻³. Literature survey reveals that the drug can be estimated only by HPLC⁴ and no spectrophotometric methods have been reported. The present study describes a simple, sensitive, accurate and precise spectrophotometric method for estimation of ACX in bulk and capsule formulation. UV analysis of ACX was performed in distilled water. The spectrum was recorded from 200 nm to 400 nm. The quantitative analysis was carried out at 231 nm. The method was validated and applied for the determination of ACX in capsule dosage form.

Figure 1. Chemical structure of acipimox.

Experimental

The spectrophotometric measurements were carried out using Elico UV/Visible double beam spectrophotometer SL-164 with 1 cm matched quartz cells.

Reagents

ACX was tested for purity by measuring its melting point and IR spectra and no impurities were found. Doubly distilled water of analytical grade was used. A pharmaceutical preparation of ACX was obtained from local pharmacy.

Standard solutions

Standard stock solution of ACX ($1000 \mu\text{g mL}^{-1}$) was prepared in distilled water. It was further diluted to obtain 1, 2, 3, 4, 5, 6, 7, 8, 9 and $10 \mu\text{g mL}^{-1}$ with distilled water. The absorbance was measured at 231 nm against distilled water as blank. The calibration curve was plotted in the concentration range of 1 to $10 \mu\text{g mL}^{-1}$ of ACX in distilled water.

Sample solutions

Twenty capsules containing ACX were weighed accurately and emptied. The powder equivalent to 100 mg ACX was dissolved in 20 mL of distilled water, sonicated for 15 minutes and filtered through Whatman No. 41 filter paper. The residues were washed thoroughly with distilled water and further diluted with distilled water to $5 \mu\text{g mL}^{-1}$ concentrations and the absorbance measured at 231 nm against distilled water as a blank.

Results and Discussion

The UV spectrum of standard solutions of ACX in distilled water was illustrated in Figure 2. The optical characteristics such as Beer's law limit⁵, molar absorptivity, Sandell's sensitivity⁶, slope and intercept are summarized in Table 1. The assay and precision studies results for capsules containing ACX are shown in Table 2.

Figure 2. UV spectrum of Acipimox in distilled water.

Table 1. Optical characteristics of proposed method.

Parameters	Values
$\lambda_{\max}$, nm	231
Beer's law limit, $\mu\text{g mL}^{-1}$	1-10
Sandell's sensitivity, $\mu\text{g cm}^{-2}/0.001$ absorbance unit	9.75×10^{-5}
Molar absorptivity, $\text{L mol}^{-1} \text{cm}^{-1}$	1.5104×10^4
Regression equation, $Y = a + bc$	
	Slope (b) 0.0978
	Intercept(a) 0.0002
Correlation coefficient, r^2	0.9998

Validation

The assay of ACX was validated with respect to stability, linearity, precision and accuracy.

Stability

The standard stock solutions of ACX were stored, in two different conditions, at $+4^\circ\text{C}$ and at ambient temperature for one month. During this period, the solutions were analyzed with UV spectrophotometric method, the spectrum was compared with the spectrum of daily prepared standard solution and no difference was obtained between them. It is decided that ACX is highly stable in the mentioned conditions.

Linearity and range

In developed UV method, calibration curve was linear in the range from 1 to $10 \mu\text{g mL}^{-1}$ of ACX.

Precision

Inter-day precision

This was done by analyzing formulation by same analyst for six days subsequently. The % RSD values are shown in Table 2.

Intra-day precision

This was done by analyzing formulation in same day for six times of individual preparation and observation. The % RSD and data are shown in Table 2.

Table 2. Assay results and precision studies.

Sample	Labeled amount, mg/ capsule	Amount found in mg*	%, label claim* $\pm$ S.D	Precision**		
				Repeatability	Inter-day	Intra-day
Acipimox capsules	250	249.96	99.92 $\pm$ 0.293	0.268	0.0068	0.0043

* Average of six determinations. **SD of five determinations

Accuracy

Recovery studies were performed to judge the accuracy of the method. Recovery studies were carried out by adding a known quantity of pure drug to a pre-analyzed formulations and the proposed method was followed. From the amount of drug found, percentage recovery was calculated. The results of analysis and recovery studies are given in Table 3.

Table 3. Recovery study.

Drug	Label claim mg /capsule	Estimated amount	Spike level, %	Amount of drug added, mg	Amount of drug recovered, mg	Percentage Recovery $\pm$ SD*
Acipimox capsules	250	249.96	80	4.0	4.03	100.72 $\pm$ 0.6473
			100	5.0	4.98	99.50 $\pm$ 0.4235
			120	6.0	6.01	100.12 $\pm$ 0.2456

*Mean of six determinations

Conclusion

In this study a simple and rapid UV spectrophotometric method for the determination of ACX in bulk and pharmaceutical formulation has been developed and validated.

References

1. Ericsson S, Eriksson M and Angelin B, *Eur J Clin Invest.*, 2008, **20**, 261-265.
2. Saloranta C, Taskinen M R, Widen E, Härkönen M, Melander A and Groop L, *Diabetes*, 1993, **42**, 1559-1566.
3. Alessio D, Gabriella C, Cristiano C and Zina G, *Exp Gerontol.*, 2004, **39**, 1061-1067.
4. Musatti L, Maggi E, Moro E, Valzelli G and Tamassia V, *J Int Med Res.*, 1981, **9**, 381-386.
5. Beckett H A and Stenlake B J, *Practical Pharmaceutical Chemistry*; 4th Ed., CBS Publishers, New Delhi, 2001, 275.
6. Sandell E B, *Colorimetric determination of traces of metals*; Inter Science, New York, 1950, 29.