Trade Science Inc.

June 2009 *Volume 8 Issue 2*

Analytical CHEMISTRY

An Indian Journal

Full Paper

ACAIJ, 8(2) 2009 [176-178]

Validated HPTLC method for the simultaneous determination of diclofenac potassium and tizanidine hydrochloride in tablet dosage form

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Received: 10th April, 2009 ; Accepted: 15th April, 2009

ABSTRACT

A simple, precise, accurate and rapid High Performance Thin Layer Chromatography (HPTLC) method was developed for the simultaneous estimation of Diclofenac Potassium and Tizanidine Hydrochloride in tablet dosage forms. A CAMAG Linomat IV Sample Applicator in Precoated silica gel Plate 60F 254 (Merck) and the mobile phase solvent system consisting of Toluene: Isopropyl alcohol: Ammonia (30:20:2.5) were used. The drugs were scanned for R_f values in the scanner at the wave length 280nm. The R_f value of Diclofenac Potassium and Tizanidine were 0.62 - 0.69 and 0.85-0.92 respectively. The Linearity for Diclofenac Potassium and Tizanidine HCL were in the range of 3000 to 7000 μg/ml and 120 to 260 μg/ml.Results of the analytical method were validated statically, and by recovery studies. The Proposed method can be successfully used to determine the drug contents of marketed formulation.

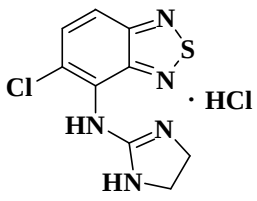
KEYWORDS

Diclofenac potassium;
Tizanidine HCl;
HPTLC.

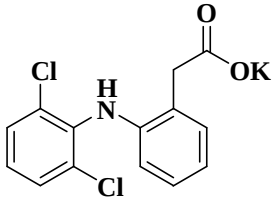
INTRODUCTION

Diclofenac Potassium and Tizanidine HCl combination is used clinically for its NSAID properties. Diclofenac Potassium is chemically 2-[(2,6-dichloro phenyl)amino]benzeneaceticacid, monopotassium salt and TizanidineHCl is 5-chloro-4-(2-imidazoline-2-ylamino)-2,1,3-benzothiodiazole hydrochloride.

The mechanism of action is not completely understood but may be related to prostaglandin synthetase inhibition. Tizanidine is an agonist at α2-adrenergic receptor sites and presumably reduces spasticity by increasing presynaptic inhibition of motor neurons. On detailed literature survey, it was found that these drugs have been estimated individually and in combination by various methods[1-11]. Besides, UV method for simultaneous estimation of this combination was reported[1] in this communication we report a new HPTLC method for simultaneous estimation of Diclofenac Potassium and Tizanidine Hydrochloride in tablet dosage forms, which is simple, rapid and precise.



Tizanidine HCl



Diclofenac potassium

EXPERIMENTAL

CAMAG Linomat IV Sample Applicator is used in Application mode and CAMAG TLC Scanner III and version 4.01 is used in scanner mode. The Plate Size of

20 x 15 cm(width x Height) Precoated silica gel Plate 60F 254 (Merck) is used as stationary phase and the mobile phase solvent system consisting of Toluene: Isopropyl alcohol: Ammonia (30:20:2.5) were used. The sample were spotted at a distance of 15mm and developed in CAMAG twin trough chamber. The detection was performed at 280nm.

Chemicals and reagents

- Isopropyl alcohol (HPLC Grade)
- Methanol (HPLC Grade)
- Toluene (AR Grade)
- Ammonia (AR Grade)
- Diclofenac Potassium (working standard)
- Tizanidine Hydrochloride (working standard)

Preparation of mobile phase

An accurately measured volume of Toluene: Isopropyl alcohol: Ammonia in the ratio of 30:20:2.5 were mixed and used as an ideal mobile phase solution.

Preparation of standard stock solution

An accurately weighed quantity of 20 mg of Tizanidine Hydrochloride and 500 mg of Diclofenac Potassium was dissolved in methanol. Make upto 10 ml to obtain a stock solution of 2000 mcg/ml of Tizanidine Hydrochloride and 50000 mcg/ml of Diclofenac Potassium.

Linearity and calibration

To evaluate the linearity range of Tizanidine Hydrochloride and Diclofenac Potassium standard stock solution was diluted with methanol to give a minimum of 5 concentrations in the range of 120-260 mcg/ml Tizanidine Hydrochloride and 3000-7000 mcg/ml of Diclofenac Potassium respectively.

Sample preparation

Twenty tablets were weighed and powdered.The powder equivalent to 20 mg of Tizanidine Hydrochloride and 50 mg of Diclofenac Potassium was weighed and transferred into a volumetric flask. Made up to 10 ml with methanol, shaked for 15 min. mixed all the contents with the aid of ultrasonicator for 1 min and filtered through Whatman filter paper 42.

S.no	Peak area of standard		Peak area of sample	
Drug	Diclofenac-potassium	Tizanidine HCl	Diclofenac-potassium	Tizanidine HCl
1	50340.1	4198.1	51025	4105.1
2	50127.1	4187.2	50304.1	4157.9
3	49987.1	4175.1	51021.1	4102.1
Avg	50151.4	4186.8	50783.4	4127.7
Std. dev	177.75357	11.50522	415.09056	31.38598
RSD	0.354433688	0.274797368	0.817374489	0.761481528

RESULTS AND DISCUSSION

Assay

The sample solution was spotted on the chromplate with the help of Linomat IV spotting system. The chromplate was developed in a twin trough chamber containing the Mobile Phase..The solvent system consisting of Toluene: Isopropyl alcohol: Ammonia (30:20:2.5) was found to be ideal mobile phase for the separation of Tizanidine Hydrochloride and Diclofenac Potassium. The well resolved bands of the drugs were scanned for R_f values in the scanner at the wave length 280nm. The R_f value was found to be between 0.62 - 0.69 and 0.85-0.92 for Diclofenac Potassium and Tizanidine Hydrochloride respectively.

Validation parameters

Linearity

Linearity was evaluated by plotting peak area as a function of analyte concentration for Diclofenac Potassium and Tizanidine Hydrochloride. From the linearity studies the specified range determined was 3000 to 7000 µg/ml for Diclofenac Potassium and 120 to 260 µg/ml for Tizanidine Hydrochloride.

Diclofenac potassium

Concentration (µg/ml)	Peak area
0	0
3000	30123.1
4000	39789.2
5000	49987.1
6000	60024.2
7000	71435

Tizanidine hydrochloride

Concentration (µg/ml)	Peak area
0	0
120	2510.1
160	3097.1
200	4198.1
240	5031.2
260	5467.5

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Repeatability

A system repeatability test was applied to a representative densitogram to check the repeatability of the proposed method. The proposed method was used to the quantitative estimation of the Diclofenac Potassium and Tizanidine Hydrochloride tablet dosage form.

Recovery data

The validity of the proposed method was verified by the recovery studies. The recovery studies were carried out and the results expressed as percentage recovery (%). The percentage recovery studies are quite optimum for the method developed.

Sample	Mg of std drug added	Mg of drug * recovered	% of recovery
Diclofenac Potassium	0.2	0.19	99.54
Tizanidine Hydrochloride	5	4.63	99.33

***Mean of 5 values**

Statistical validation

Drug	Lable claim (mg/tablet)	Amount estimated (mg/tablet)	Std dev	%rsd	Std error
Diclofenac potassium	50	9.85	0.08	0.80	±0.04
Tizanidine hydrochloride	2	1.99	0.03	0.30	±0.01

CONCLUSION

In this present study an attempt has been made to develop an analytical method for the simultaneous estimation of Diclofenac Potassium and Tizanidine Hydrochloride in combined tablet dosage form. The present combination of Diclofenac Potassium and Tizanidine Hydrochloride was marketed as one formulation. Diclofenac Potassium-50mg /tab

Tizanidine Hydrochloride-2mg/tab. The fixed dose combination tablet of Diclofenac Potassium and Tizanidine Hydrochloride was subjected to simultaneous estimation by HPTLC method. The proposed HPTLC method was validated by evaluation of the validation parameters. The Linearity parameter, Repeatability recovery studies ,statistical validation and assay Parameters were performed with in a short analysis time. Highly reliable HPTLC method was developed for the quantitative estimation of Diclofenac Potassium and Tizanidine Hydrochloride in combined tablet dosage form.

The results obtained were reproducible and reliable. The validity and Assay of the methods were evident from the statistical and analytical parameters obtained.

From the forgoing it is concluded that the methods developed are simple, rapid, selective and precise hence suitable for application in routine analysis of pharmaceutical preparations.

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