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Research Article

Development of a spectrophotometric method for the assay of Diclofenac potassium

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Abstract

Diclofenac potassium is a non- steroidal anti-inflammatory drug and a prostaglandin (PG) synthetase inhibitor. The objective of this study was to develop a spectrophotometric method for the assay of diclofenac potassium. A series of diclofenac potassium solutions were prepared ranging from 200 ppm to 12.5 ppm. After preparation of a standard stock solution of 200 ppm in 100 ml of water, different dilutions were made (100ppm, 50ppm, 25ppm and 12.5ppm). At the wavelength 242nm, the absorbance of the standard preparation and dilutions was measured using a UV spectrophotometer. In this study a precise and accurate method was developed. The validation of the developed method for the assay of diclofenac potassium was done by various parameters that included linearity, accuracy test and precision. The method showed good reproducibility and good recovery with % RSD less than 2. The method was found to be simple, economical, rapid, specific, precise and accurate.

Key words: Diclofenac Potassium, accuracy, linearity, regression equation, precision

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1. Introduction

Chemically diclofenac potassium is a monobasic potassium salt {2-[(2,6-Dichlorophenyl)amino]-benzene acetic acid} . as shown in Figure 1. . It belongs to a class of non-steroidal anti-inflammatory drugs (NSAIDs), and is effective against analgesia, fever and inflammation [1]. It is classified under prostaglandin (PG) synthetase inhibitors [2-4]. Diclofenac potassium acts by inhibiting cyclooxygenase (COX) enzyme, the enzyme responsible for the formation of prostaglandins (PGs) such as $PGF_2\alpha$. The drug alleviates dysmenorrheic pain and no side effects have been reported [5]. It also restores exercise performance in women suffering from dysmenorrhea [6].

A method for the determination of diclofenac potassium individually or in combination with other drug preparations

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by High-performance liquid chromatography (HPLC) has been reported by Khatal LD et al [7]. A UV spectrophotometric method for the estimation of diclofenac potassium in a formulation has also reported [8]. Other assay methods for the determination of diclofenac potassium were High-performance liquid chromatography (HPLC) method [9-11] and UV-spectroscopy method [12-13]. The present work describes the development of a validated UV-spectroscopy method that can quantify diclofenac potassium from a dosage form. The present diclofenac potassium UV-spectroscopy method was validated following the International Conference on Harmonisation guidelines.

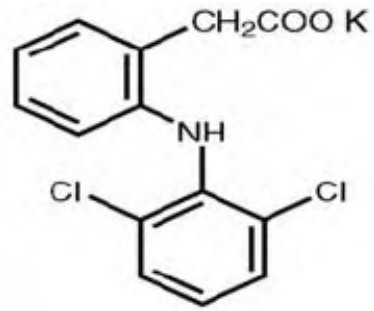

Figure1: Structure of diclofenac potassium

2. Experimental Design

Materials and Methods: A standard diclofenac potassium sample was supplied by Abbot Pharmaceutical Laboratories (Pakistan limited) in bulk.

Instrumentation

UV visible spectrophotometer Shimadzu double beam 1601 was used to measure spectra. The solvent used was water for the assay.

Wavelength Selection

A series of diclofenac potassium solutions were prepared ranging from 200 ppm to 12.5 ppm accurately by dissolving the active diclofenac potassium in water. The diclofenac potassium solution was scanned in the UV regions. The wavelength maximum (λmax) was observed at 242nm.

Procedure

A series of diclofenac potassium solutions were prepared ranging from 200 ppm to 12.5 ppm. After preparation of a standard stock solution of 200 ppm in 100 ml of water, different dilutions were made (100ppm, 50ppm, 25ppm and 12.5ppm). At the wavelength 242nm, the absorbance of the standard preparation and dilutions was measured using the UV spectrophotometer. The quantity in mg, of diclofenac potassium was determined.

3. Results and Discussion

Method validation

The validation of developed method for the assay of diclofenac potassium was done by various parameters that included linearity, accuracy test and precision.

Linearity

Linearity was determined in the range of 12.5-200 μg mL^{-1}. Least square linear regression analysis was applied to Peak area versus concentration of diclofenac potassium. A linear regression line was obtained with correlation coefficient (R2 > 0.9993). The regression equation for active (what is active here?) is displayed in Figure 2.

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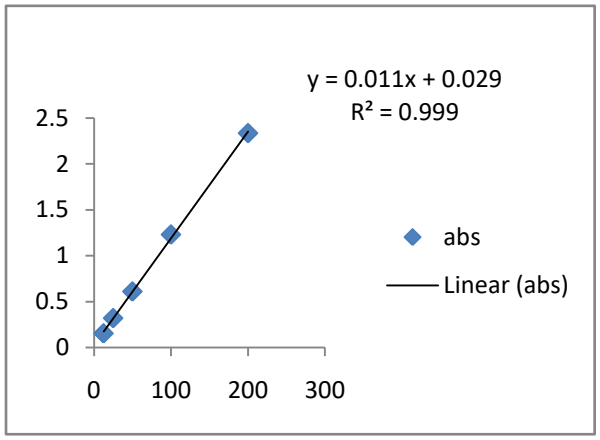
Accuracy

Accuracy of the developed method for the



assay of diclofenac potassium was determined as the percentage of recovery of known amounts of diclofenac potassium at spike concentration that was 40, 50 and 60 mg mL−1. Every sample was taken five times and result range was 99.8%-100.20%, (Table 2)

The method has a high degree of accuracy and this was indicated by high recovery.

Figure 2: Linearity of diclofenac potassium

Precision

Repeatability (i.e. intra-day precision and inter day precision) of the developed method for the assay of diclofenac potassium was determined. It was defined as relative standard deviation % RSD. Five different dilutions of diclofenac potassium (200ppm, 100ppm, 50ppm, 25ppm and 12.5ppm) in the linear range were analyzed in the same day (intra-day precision) and two consecutive days (inter-day precision); every sample was taken five times. Good precision was observed in both intra and inter-day %RSD values, the values were in the range of 0.51-1.92% (Table 3).

Diclofenac potassium	**absorbance**
200	2.334
100	1.23
50	0.61
25	0.32
12.5	0.154

Table No. 1 : Absorbance of drug in different concentration

Accuracy			
Drugs	Conc	%RSD	% Recovery
Diclofenac potassium	80%	1.20	100.20
	100%	1.20	100.00
	120%	1.30	99.98

Table 2: Accuracy of diclofenac potassium

The insignificant results showed no remarkable difference in inter and intra-day precision.

4. Conclusion

In concentration range of 12.5-200 µg/ml the calibration curve was linear for diclofenac potassium with correlation coefficient of 0.9993. The precision both intra and inter-day and accuracy of the proposed method was determined and validated statistically. The method showed good reproducibility and good recovery with % RSD less than 2. The method was found to be simple, economical, rapid, specific, precise and accurate. This method can be successfully applied for the routine analysis of diclofenac potassium. By performing accuracy studies, the accuracy of the proposed method was confirmed, and the results were acceptable. The precision of the proposed method was confirmed by performing intraday and inter day precision. The method for the determination of diclofenac potassium was validated according to ICH guidelines. This

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method can be adapted for routine assay. The intra-run and inter-run variability and accuracy results were in the acceptable limit according to ICH guidelines. The short analysis time (< 5min) enables its application in routine and quality control analysis of finished products.

Inter day and intraday precision of diclofenac potassium					
	Conc. ug mL^{-1}	**Inter-day**		**Intra-day**	
Drugs		%RSD	%Recovery	%RSD	%Recovery
Diclofenac potassium	200	0.55	101.07	0.51	98.00
	100	1.26	102.24	1.2	100.00
	50	1.91	100.56	1.92	100.00
	25	1.79	101.04	1.72	100.40
	12.5	1.12	100.65	1.11	100.40

Table No. 3: Precision of Diclofenac Potassium

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