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METHOD DEVELOPMENT AND VALIDATION OF DICLOFENAC SODIUM BY USING UV SPECTROSCOPY

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ABSTRACT

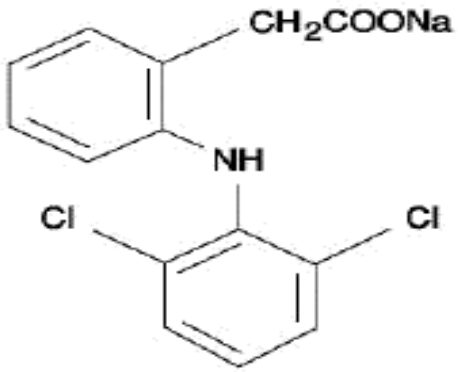
A simple, selective, accurate, precise spectroscopic method for the estimation of diclofenac sodium in bulk and pharmaceutical tablet dosage form has been developed and validated. The linearity range of diclofenac is 10-30µg/ml. The LOD and LOQ were found to be 1.03g/ml and 3.12µg/ml respectively. The amount of diclofenac was calculated as 99.42%. Further the precision of the method was confirmed by the repeatable analysis of solution. The % RSD was found to be 1.370 it indicated that the method has good precision. The % recovery was found to be in the range of 98.72-99.15%. the % recovery was calculated for 80%, 100% and 120% RSD value indicated that there is no interference due to excipients used in formulation. Hence the accuracy of the method was confirmed.

KEYWORDS: Diclofenac sodium, UV method, validation.

INTRODUCTION

Diclofenac sodium is chemically sodium salt of 2-[{2,6-dichloroanilino)phenyl] acetic acid. moreover it is having anti inflammatory, antipyretic and analgesic properties, this leads to an inhibition of prostaglandins that are involved in and fever. literature survey revealed several analytical

methods like UV spectrophotometry and HPLC, these have been reported in bulk, pharmaceutical dosage form for the determination of diclofenac sodium. It is used for treatment of rheumatoid arthritis, ankyllosing spondylitis, osteoarthritis and sport injuries. To our noticed reviewed for method development and validation of diclofenac sodium by using uv spectroscopic method for good accuracy, linearity and precision.



Diclofenac sodium

IUPAC name: [2-(2,6-dicloroanilino)phenyl]acetic acid.

chemical formula: C14H10Cl2NNaO2

Molecular weight: 318.129gm/mol

MATERIALS AND METHODS

INSTRUMENTATION

Instruments for measuring the absorption of U.V. or visible radiation are made up of the following components;

1. Radiation source: It is important that the power of radiation source does not change abruptly over its wavelength range. both deuterium and hydrogen lamps emit radiation in the range of 160-375nm. quartz windows must be used in these lamps, and quartz cuvettes must be used, because glass absorbs radiation of wavelengths less than 350nm.

2. Filters or monochromators: All monochromators contain the following component parts;

a) An entrance slit

b) A collimating lens

c) A dispersing device(a prism or a grating)

d) A focusing lens

e) An exit slit

3. Sample containers or sample cells: Quartz or fused silica cuvettes are required for spectroscopy in the UV region. silicate glasses can be used for the manufacture of cuvettes for use between 350 and 2000nm.

4. Detectors: In order to detect radiation, here 3 types of photosensitive devices are

a) Photo voltaic cells or otherwise called it as barrier - layer cell.

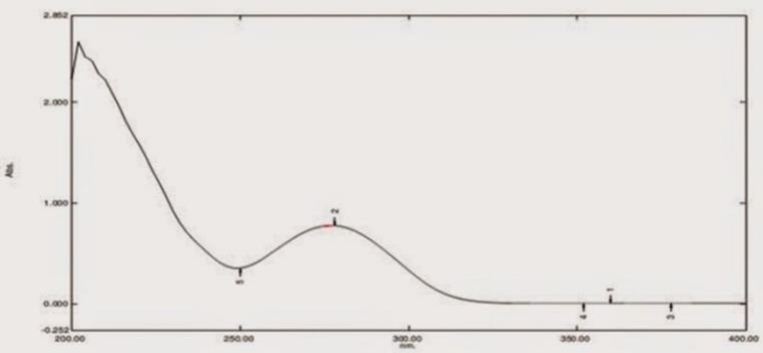
b) Photo tubes or photoemissive tubes.

c) Photo multiplier tubes.

Method development

Determination of UV wavelength range

For the determination of analytical UV wavelength method 20µg/ml drug solution of diclofenac sodium was scanned in the spectrum mode from 400nm-200nm against distilled water as blank. The standards were prepared between concentrations of 10, 15, 20, 25, 30µg/ml. wavelength range was selected around wavelength maxima (274nm). The final wavelength range between 274nm-300nm was selected.



UV SPECTRUM OF DICLOFENAC SODIUM

Preparation of standard stock solution

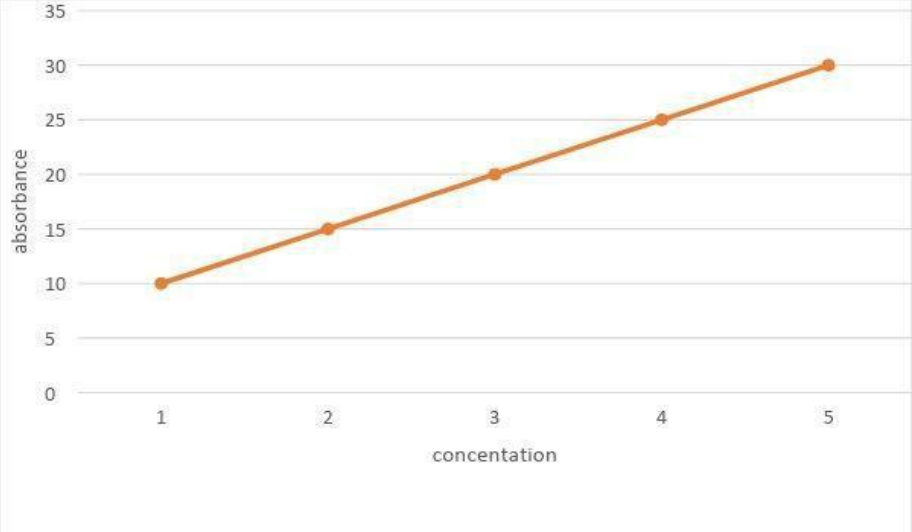
The standard stock solution of diclofenac sodium was prepared by taking 10mg diclofenac and weighed, then transfer to 100ml volumetric flask. It was dissolved in 100ml of acetic buffer and sonicated for 15mins to get homogenous solution. Then it was filtered through a 0.45µ whattman filter paper. A final concentration of 100mg/ml of diclofenac was prepared. This solution was filtered through filter paper to remove some unwanted excipients. After filtration, from this 2ml was taken and diluted to 10ml with distilled water which gives 20µg/ml solution, and the absorbance of the solution was measured at 276.20nm.

Calibration curve for diclofenac sodium

Accurately weigh and transfer the standard stock solutions to get concentrations of 10, 15, 20, 25, 30µg/ml respectively. These solutions were scanned from 400-200nm and was integrated in the rang e of 274-300nm. The calibration curve was plotted between absorbance and concentration.

S.NO	CONCENTRATION(µg/ml)	ABSORBANCE
1.	10	0.3219
2.	15	0.4888
3.	20	0.6509
4.	25	0.8186
5.	30	0.9659

CALIBRATION DATA OF DICLOFENAC SODIUM



$y = 0.032x + 0.002$

LINEARITY OF CALIBRATION CURVE

ASSAY

Twenty tablets were weighed accurately and powdered. Powder equivalent to 10mg diclofenac was weighed and transfer it into a 100ml volumetric flask. It was dissolved in a 100 ml acetic buffer and sonicated for 15mins to get homogenous solution. then it was filtered through a 0.45µ whattman fliter paper. A final concentration of 100mg/ml of diclofenac was prepared. This solution was filtered through filter paper to remove unwanted remove substances. After flitration, from this 2ml was taken and diluted to 10ml with distilled water which gives 20µg/ml solution and the absorbance of the solution was measured at 276.20 nm.

VALIDATION METHOD

1. Precision: The precision (measurement of intraday, interday, repeatability) results showed good reproducibility with percent relative standard deviation (%RSD) was below 2.0%. This indicated that method was highly precise.

CONCENTRATION	AVERAGE	SD	%RSD
15µg/ml	0.4822	0.0049	1.0185
20µg/ml	0.6488	0.0032	0.4997
30µg/ml	0.8183	0.4997	0.3484
MEAN%RSD	0.662		

INTRA DAY PRECISION

CONCENTRATION	AVERAGE	SD	%RSD
15µg/ml	0.4589	0.0038	0.8319
20µg/ml	0.6191	0.0055	0.9079
25µg/ml	0.7830	0.0028	0.4342
MEAN%RSD	0.724		
	INTERDAY PRECISION		

2. Accuracy: The accuracy for the analytical method for diclofenac was determined at 80%, 100%, 120% levels of standard solution. 191absorbance was measured at276.20nm and results were expressed in terms of % recoveries. Standard deviation and % RSD was calculated, the results are tabulated.

TEST (µg/ml)	ACCURACY LEVEL	AMOUNT OF STANDARD DRUG ADDED(µg/ml)	% RECOVERY	STANDARD DEVIATION	%RSD
20µg/ml	80%	16	98.72	0.0800	0.513
	100%	20	99.36	0.0673	0.649
	120%	24	99.15	0.0978	0.773
		%RECOVERY STUDY			

3. Linearity: The linearity of this method was determined at ranging from 10-30µg/ml for diclofenac. The regression equation was found to be y=0.0324x+0.0021 and the coefficient (r2) 0.9995. As shown in calibration curve.

4. Repeatability: The repeatability or reproducibility of 20µg/ml of concentration was taken with respect to concentration, absorbance and % RSD values.

CONCENTRATION	ABSORBANCE	%RSD
	0.6336	
	0.6398	
20µg/ml	0.643	
	0.6397	1.37
	0.636	
	0.638	
AVERAGE	0.6386	
SD	0.0088	
	REPEATABILITY	

5. Robustness: It is a study of small but deliberate variations in method parameters such as absorption maxima, Ph and ratio of mobile phase solvents. In this present work the absorption maxima was decreased and increased 2nm and carried the process for 30µg/ml solution for 6 times. The RSD % was calculated.

CONCENTRATION	AT 274.20 (-2nm)	AT278.20 (+2nm)	%MEAN RSD
MEAN	0.6336	0.6317	
SD	0.00537	0.0061	0.9204
%RSD	0.8487	0.9689	
	RESULT OF ROBUSTNESS		

6. Limit of detection(LOD): It is the smallest quantity of an analyte that can be detected and not necessarily determined, in a quantitative fashion. It was calculated by the following formula;

LOD=3.3*S.D/slope

Where, S.D= standard deviation

7. Limit of quantitation(LOQ): It is the lowest concentration of an analyte in a sample that may be determined with acceptable accuracy and precision. It was calculated by the following formula;

LOQ=10S.D/slope.

CONCLUSION

The proposed method is simple, accurate, precise and selective for the estimation of diclofenac sodium in bulk and pharmaceutical dosage forms. The method is economical, rapid and do not require any sophisticated instruments contrast to chromatographic method. Hence it can be effectively applied for the routine analysis of diclofenac in bulk and marketed tablet formulation.

RESULTS AND DISCUSSIONS

S.NO	PARAMETERS	RESULTS
1.	Absorption maxima(nm)	276.20
2.	Beers range(µg/ml)	10-30µg/ml
3.	Standard regression equation	y=0.0324x+0.0021
4.	Concentration coefficient(r2)	0.9995
5.	%Assay	99.42±0.080
6.	Precision (%RSD)	
	Intraday precision	0.662
	Interday precision	0.724
7.	Accuracy	99.72±0.080
8.	Robustness	0.9204
9.	LOD	1.03
10.	LOQ	3.12

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