Academic Sciences International Journal of Pharmacy and Pharmaceutical Sciences

ISSN- 0975-1491 Vol 5, Issue 3, 2013

Research Article

ANALYTICAL METHOD DEVELOPMENT AND VALIDATION OF ACECLOFENAC IN PHARMACEUTICAL DOSAGE DORM BY UV SPECTROSCOPY TECHNIQUE

SUSMITHA A[1]*, HEPCY KALARANI D[1], VENKATESH P[1], RAVINDRA REDDY K[1]

[1]P.Rami Reddy Memorial College of Pharmacy, Kadapa-516003, Andhra Pradesh, India. Email: aggarapusuma@gmail.com

Received: 28 Dec 2012, Revised and Accepted: 28 Mar 2013

ABSTRACT

Objective: The present work is aimed to develop a simple, accurate, precise and cost effective uv-spectroscopic method for the estimation of Aceclofenac, a non-steroidal anti inflammatory drug with good analegesic anti rheumatic activity in pharmaceutical dosage forms.

Method: The drug was dissolved in ethanol solvent. The method was validated for recovery studies and repeatability studies.

Results: The λ_{max} or absorption maxima of the drug was found to be 277.7nm. A linear response was observered in the range of 2-60µg/ml. The percentage recovery of Aceclofenac was found to be 99%. The percentage deviation ranges from 0.3 to 1.7.

Conclusion: The proposed method can be used for the estimation of Aceclofenac in quality control of formulation without interference of the excipients.

Keywords: Aceclofenac, Ethanol, UV spectroscopic technique, Non-steroidal anti inflammatory drug.

INTRODUCTION

Spectroscopy[1]

Spectroscopy is the branch of science dealing with the study of interaction of electro-magnetic radiation with matter. The most important consequence of such interaction is that energy is absorbed or emitted by the matter in discrete amounts called quanta. The absorption or emission processes are known throughout the electro-magnetic spectrum ranging from the gamma region to the radio region. The data that is obtained from spectroscopy is called a spectrum. A spectrum is a plot of the intensity of energy detected versus the wavelength (or mass or momentum or frequency, etc.) of the energy.

A spectrum can be used to obtain information about atomic and molecular energy levels, molecular geometries, chemical bonds, interactions of molecules, and related processes. Often, spectra are used to identify the components of a sample (qualitative analysis). Spectra may also be used to measure the amount of material in a sample (quantitative analysis).

Analytical Method Validation[2]

Analytical Method Validation is "the collection and evaluation of data, from the process design stage throughout production, which establishes scientific evidence that a process is capable of consistently delivering quality products".

Validation is an act of proving that any procedure, process, equipment, material, activity or system performs as expected under given set of conditions and also give the required accuracy, precision, sensitivity, ruggedness, etc.

When extended to an analytical procedure, depending upon the application, it means that a method works reproducibly, when carried out by same or different persons, in same or different laboratories, using different reagents, different equipments, etc.

Advantages of Analytical Method Validation

- The biggest advantage of method validation is that it builds a degree of confidence, not only for the developer but also to the user.
- Although the validation exercise may appear costly and time consuming, it results inexpensive, eliminates frustrating repetitions and leads to better time management in the end. Minor changes in the conditions such as reagent supplier or grade, analytical setup are unavoidable due to obvious reasons but the method validation absorbs the shock of such conditions and pays for more than invested on the process.

Key Parameters of the Analytical Method Validation[2]

Validation is a process that consists of at least four distinct steps

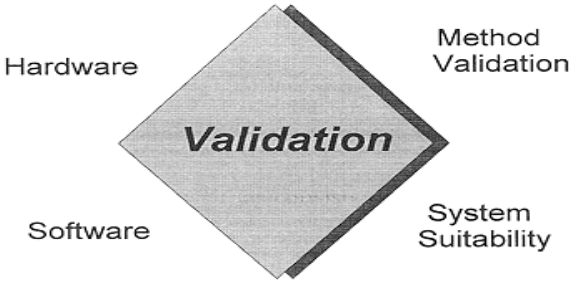
(1) Software validation,

(2) Hardware (instrumentation),

(3) Method validation,

(4) System suitability,

The process begins with validated software and a qualified system



- Accuracy
- Precision
- Linearity and range
- Limit of detection
- Limit of quantification
- Selectivity and Specificity
- Robustness
- Ruggedness
- Stability and System suitability tests.

Aceclofenac is chemically 2-[2-[2-[(2,6-Dichloro phenyl)amino]phenyl]acetyl]oxyacetic acid which is a Non-steroidal anti inflammatory drug, it is a potent inhibitor of the enzyme cyclooygenase which is involved in the production of prostaglandins.

A detailed survey of literature revealed UV-spectrophotometric methods reported for the simultaneous estimation of Aceclofenac and Rabeprazole sodium from the combined capsule dosage form[3], Aceclofenac and Tizanidine in bulk drug and tablet formulations[4], but no method was reported for the estimation of Aceclofenac in its tablet dosage form using Ethanol as a solvent. In the present investigation, an attempt was made to develop a simple and precise ultra violet (UV) spetrophotometric method with greater precision, accuracy and sensitivity for the analysis of Aceclofenac in its pharmaceutical dosage forms.

MATERIALS AND METHODS

Instruments and Reference standard

An UV- visible double beam spectrophotometer-Systronics 2203(smart) with Matched quartz cells corresponding to 1 cm path length.GE 412 Single pan electronic balance was used for weighing the materials. Pure samples of Aceclofenac were obtained from biochem, Hyderabad, India. Ethanol of analytical grade was purchased from E.Merck (India) Ltd., Mumbai, India.

Trade Name	Company Name	Dose	Batch Number	Manufactured Date	Expiry Date
Aceclo Tab	Aristo	100mg	340F012	June 2012	May 2014
Aceclo plus Tab	Aristo	600mg	375F112	Oct 2012	Sep 2013

Procedure

Preparation of standard stock solutions

The standard stock solution of drug was prepared by dissolving 50mg of the drug in 50 ml standard flask using Ethanol as a solvent to give a concentration of 1000 µg/ml. This stock solution on further dilutions is used for establishing following parameters.

Concentration of solvent and Wavelength selection

Solutions of concentration of 10 µg/ml, 100µg/ml were prepared. They were subjected to scanning from 200-400nm.From the different absorbance values obtained 277.7nm was selected for the present work.

Beer's law range

The stock solution was suitably diluted with Ethanol to get concentration range from 1 to 1000 µg/ml. The solutions are scanned in UV regions between 200 to 300nm the absorption were measured at λ_{max} found. Using absorbance values against concentrations plotted the calibration curve and the linearity range can be found.

Analysis of Tablet Formulations

Twenty tablets were finely powdered. An accurately weighed quantity of powder equivalent to about 100mg of Aceclofenac was transferred to a 100ml standard flask. The contents of the flask was mixes with ethanol and shaken to dissolve the active ingredients and then made up to the volume with the same solvent. The solution was filtered and the filtrate was further diluted with ethanol to give a final drug concentration of 6 to 30µg/ml. Absorbance values of sample solution were recorded at 277.7nm.

The proposed method is validated for the following parameters.

a. Recovery studies

b. Repeatability studies

Recovery Studies

The accuracy, precision, suitability and validity of the proposed method were satisfied by conducting percentage recovery studies. They were carried out by adding a known quantity drug to the pre-analyzed sample and the contents were reanalyzed by proposed method.

$$\text{Percentage recovery} = \frac{\text{Amount of drug found in the sample after addition of drug -- amount of drug found in the sample}}{\text{Amount of standard drug added}}$$

Repeatability Studies

Repeatability expresses the precision under the same operating conditions. It is also termed as intra assay precision. It is assessed by using as minimum of 9 determinations for each table covering the specified range for the procedure (3 concentrations per 3 replicates each) [5,6].

RESULTS AND DISCUSSION

- The UV spectra of Aceclofenac were presented. The absorption maxima was observed at 277.7nm. Obeyance to beers law was confirmed by the linearity of the calibration curve of Aceclofenac. Aceclofenac showed linearity in the concentration range of 2-60 µg/ml. Linearity data was given in table 1 and the curve was shown in fig 1.
- The quantitative estimation was carried out in tablet formulations by taking concentrations of 6 – 30 µg/ml. The brands of formulations shows the percentage purity values range from 99.2 to 101% the percentage deviation values were found to be between – 0.3 to +1.7, and the values shown in table 2 and 3.
- The quantitative results obtained were subjected to statistical analysis to find out standard deviation and standard error values. The relative standard deviation values are below 2%, indicating the precision of the methodology and low standard error values show the accuracy of the method and the values were shown in tables 4 and 5.

Table 1: Linearity data

S. No.	Concentration(µg/ml)	Absorbance
1.	1	0.040
2.	2	0.141
3.	3	0.203
4.	4	0.234
5.	5	0.286
6.	6	0.323
7.	7	0.398
8.	8	0.445
9.	9	0.498
10.	10	0.544
11.	20	0.999
12.	30	1.259
13.	40	1.464
14.	50	1.574
15.	60	1.580
16.	70	1.586

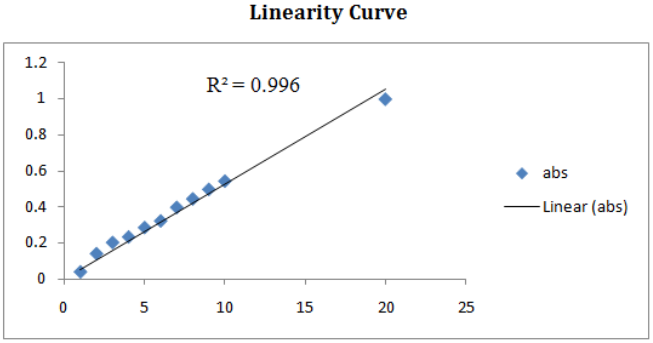


Fig. 1: Calibration curve of aceclofenac

Table 2: Quantitative estimation of aceclofenac

Formulation - I

S. No.	Concentration (µg/ml)	Label claim (mg)	Amount present (mg/tab)	Percentage Label claim (%w/w)	Percentage Deviation (%w/w)
1.	6	100	99.7	99.7	±0.30
2.	8	100	100.8	100.8	±0.80
3.	10	100	101.1	101.1	±1.1
4.	20	100	99.8	99.8	±0.2
5.	30	100	100.3	100.3	±0.3

Table 3: Quantitative estimation of aceclofenac

Formulation- II

S. No.	Concentration (µg/ml)	Label claim (mg)	Amount present (mg/tab)	Percentage Label claim (%w/w)	Percentage Deviation (%w/w)
1.	6	600	599	99.8	±0.2
2.	8	600	601	100.1	±0.1
3.	10	600	603	100.5	±0.5
4.	20	600	598	99.6	±0.4
5.	30	600	600	100.1	±0.1

Table 4: Quantitative estimation of aceclofenac

Statistical data of formulation - I

S. No.	Concentration (µg/ml)	Percentage Label Claim (%w/w)	Standard Deviation (S.D)	Relative standard Deviation (R.S.D)	Standard Error of mean (S.E)
1.	6	99.7	0.0004	0.004008	0.0001789
2.	8	100.8	0.0008	0.002004	0.0000447
3.	10	101.1	0.009	0.002004	0.0000447
4.	20	100.2	0.0007	0.002004	0.0000447
5.	30	100.3	0.0002	0.002004	0.0000447

Table 5: Quantitative estimation of aceclofenac

Statistical data of formulation-II

S. No.	Concentration (µg/ml)	Percentage Label Claim (%w/w)	Standard Deviation (S.D)	Relative standard Deviation (R.S.D)	Standard Error of mean (S.E)
1.	6	100.2	0.0014	0.0023	0.0006261
2.	8	99.8	0.0021	0.0034	0.0009392
3.	10	100.4	0.0011	0.0018	0.0004914
4.	20	100.1	0.009	0.00149	0.004025
5.	30	99.3	0.007	0.00130	0.004017

Repeatability Studies

- The repeatability of the method was confirmed by the assay procedures with 3 different concentrations of 3 replicates each. The results obtained in repeatability test expresses the precision of the given method. And the values were shown in table 6 and 7.

Recovery Studies

- The validation of the proposed method was further confirmed by recovery studies. The recovery values vary from 98.54 to 100.70% w/w. This serves as a good index of accuracy and reproducibility of the study and the values were shown in tables 8 and 9.

Table 6: Validation of aceclofenac

Repeatability studies of formulation-I

S. No.	Concentration (µg/ml)	Label claim (mg)	Amount present (mg)	Percentage Label claim (%w/w)	Percentage deviation (%w/w)
1.	6	100	99.77	99.7	±0.3
2.	6	100	99.6	99.6	±0.4
3.	6	100	99.7	99.7	±0.3
4.	8	100	101.0	101.1	±1.1
5.	8	100	101.3	101.3	±1.3
6.	8	100	101.2	101.2	±1.2
7.	10	100	101.1	101.1	±1.1
8.	10	100	101.3	101.3	±1.3
9.	10	100	101.7	101.7	±1.7

Table 7: Validation of aceclofenac

Repeatability studies of formulation-II

S. No.	Concentration (µg/ml)	Label claim (mg)	Amount present (mg)	Percentage Label Claim (%w/w)	Percentage deviation (%w/w)
1.	6	600	596.3	99.3	±0.7
2.	6	600	598.1	99.6	±0.4
3.	6	600	601.9	100.1	±0.1
4.	8	600	604.6	100.6	±0.4
5.	8	600	604.6	100.6	±0.4
6.	8	600	603.9	100.5	±0.5
7.	10	600	599.0	99.8	±0.2
8.	10	600	606.4	101.1	±1.1
9.	10	600	606.2	101.1	±1.1

Table 8: Validation of aceclofenac

Recovery studies of formulation-I

S. No.	Concentration (µg/ml)	Amount Of Standard Drug Added (mg)	Amount Recovered (mg)	Percentage Recovered (%w/w)	Percentage Deviated (%w/w)
1.	6	10	109.9	109.9	±0.1
2.	8	10	109.8	109.8	±0.2
3.	10	10	109.6	109.6	±0.4
4.	20	10	110.2	110.2	±0.2
5.	30	10	110.1	110.1	±0.1

Table 9: Validation of aceclofenac

Recovery Studies of formulation-II

S. No.	Concentration (µg/ml)	Amount of Standard Drug Added (mg)	Amount Recovered (mg)	Percentage Recovered (%w/w)	Percentage Deviated (%w/w)
1.	6	10	610.1	109.8	±0.2
2.	8	10	609.2	109.0	±0.1
3.	10	10	609.3	109.5	±0.5
4.	20	10	610.4	109.7	±0.3
5.	30	10	609.9	109.6	±0.4

CONCLUSION

The proposed method of analysis is novel, simple, cost-effective, safe, accurate and reproducible. This method can be routinely employed in the analysis of Aceclofenac in tablet formulations precluding using Ethanol as a solvent.

ACKNOWLEDGEMENT

The authors are grateful to Biochem, Hyderabad, India for providing gift samples of Aceclofenac. The authors are also grateful to P.R.R.M College of pharmacy, Kadapa for providing necessary facilities to carry out this work.

REFERENCES

1. Introduction to Spectroscopy- http://www.wikipedia.com/ web dictionary.
2. J.Ermer & J.H.McB.Miller-Method Validation in Pharmaceutical Analysis- P.21-171
3. Ramesh LS, Sanket DH, Ganesh KD, Charusheela AB, Pravin ST- Spectrophotometric methods for simultaneous estimation of Rabeprazole sodium and Aceclofenac from the combined capsule dosage form- Pharmaceutical Methods- A Pharmaceutical Journal of Analysis- Pharm Methods [serial online] 2011 [cited 2012 Nov23];2:1937. Available from: http://www.phmethods.org/text.asp?2011/2/3/193/90362.
4. Gondane S.J, Deshpande M.M., Mahajan. M.P., Sawant S.D - Spectrophotometric method development and validation for estimation of Tizanidine and Aceclofenac in Bulk Drug & Tablet formulation- International Journal of ChemTech Research- - Vol.3, [updated 2011 Apr; cited 2011 Jun]
5. Neetu Sachan*, Phool Chandra, Mayank Yadav-Simple and validated uv-spectrophotometric method for the estimation of Levofloxacin in bulk and pharmaceutical dosage forms- International Journal of Pharmacy and Pharmaceutical Sciences-2012; Vol 4, Suppl 5.
6. Lavanya gogulamudi* and Sujana. K- Development and validation of uv-spectroscopic method for determination of Tramadol Hydrochloride in bulk and formulation- International Journal of Pharmacy and Pharmaceutical Sciences- 2012; Vol 4, Suppl 5.