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NOVEL RP-HPLC AND FT-IR METHODS FOR THE ESTIMATION OF RELATED SUBSTANCES AND QUANTITATION OF ACECLOFENAC

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ABSTRACT

Aceclofenac is a phenyl aceticacid derivative with potent analgesic and anti inflammatory properties and an improved gastro intestinal tolerance. In the present study, a Reverse Phase HPLC method and FTIR methods were developed for the analysis of Aceclofenac for related substances and concordance with the standard. Both the methods showed excellent sensitivity with 0.008% total impurities and the sample spectrum is 99.89% concordant with that of standard aceclofenac . This sensitive and selective method can be used for detection of related substances in aceclofenac in order to control and regulate process related impurities.

Keywords *– RP-HPLC, FT-IR, Aceclofenac, Related Substances, Concordance.*

1. INTRODUCTION

Aceclofenac [(2,6-dichlorophenylamino)phenyl]acetoxyacetic acid is a phenyl acetic acid derivative that shows analgesic properties and good tolerability profile in a variety of painful conditions. It is a Non Steroidal Anti Inflammatory Drug(NSAID) indicated in the symptomatic treatment of pain and inflammatory or degenerative orthopathies such as osteoarthritis, Rheumatoid arthritis, Ankylosing, Spondilytis, Abarticular inflammations, Post Trauma or Post operative inflammations[1].

The presence of minute quantities of unwanted chemicals in pharmaceutical substances which may influence the drug safety and efficiency and is a major concern in the overall quality of pharmaceuticals[2,3].

2. MATERIALS AND METHODS

2.1 *Instrument / Equipment*

a) Chromatography was performed with Waters 2489 HPLC system connected with a UV-VIS detector controlled with Empower 2 software (Waters).

The separation of analytes was accomplished using C18 Intersil Column(250mm x4.6mm id) maintained at room temperature. Final Chromatographic conditions involved an isocratic elution, using mobile phase Acetonitrile: Methanol: Water(60:30:10) and pH of final mobile phase was adjusted to 7.0 with Glacial acetic acid and Sodium Hydroxide. The pump flow rate was 0.5ml/min

b) Infra Red (IR) spectra were recorded using KBr disk on Shimadzu (8400S) spectrophotometer.

2.2 ***Chemicals***

All the chemicals were of either Analytical or HPLC grade obtained from SumagesPharmaPvt Ltd, Bhimavaram, A.P,India.

2.3 ***Related Impurities in Aceclofenac***

2.3.1 ***Mobile Phase***

The mobile phase consisting of Acetonitrile: Methanol: Water (60:30:10) % V/V was prepared and sonicated for 20minutes. The mobile phase was filtered through 0.45µ membrane filter.

2.3.2 ***Standard Stock and Sample solutions :***

Standard Stock solution Aceclofenac and sample of Aceclofenac(No. 1412120) 1mg//1ml each were individually prepared by dissolving 10mg each in the mobile phase separately in 10ml Vol flasks. The volume of 1ml of above solutions were diluted to 10ml with mobile phase separately in volumetric flasks (100µg/ml each).

2.4 Concordance in FTIR

Translucent pellets were prepared by dilution of Aceclofenac reference and sample substance in potassium bromide to obtain 250mg of total weight.

3. RESULTS AND DISCUSSIONS

3.1 RP-HPLC Method

The RP-HPLC Method was developed for the estimation of related impurities in Aceclofenac. The separation of analytes was carried out using C18 Intersil Column maintained at room temperature using Acetonitrile : Methanol : Water (60:30:10) as isocratic mobile phase at pH 7.0. Representative chromatogram obtained by the described method for the standard Aceclofenac and its impurities is show in figures 1and 2. The retention time for Aceclofenac, impurities were std-4.8min, sample-4.61min, impurity 1-5.01min, impurity 2-6.72min respectively and the Aceclofenac ref substance complies 99.98 % with the sample with individual impurity 0.006% and total impurity 0.008%. The results were shown in tables 1 and 2.

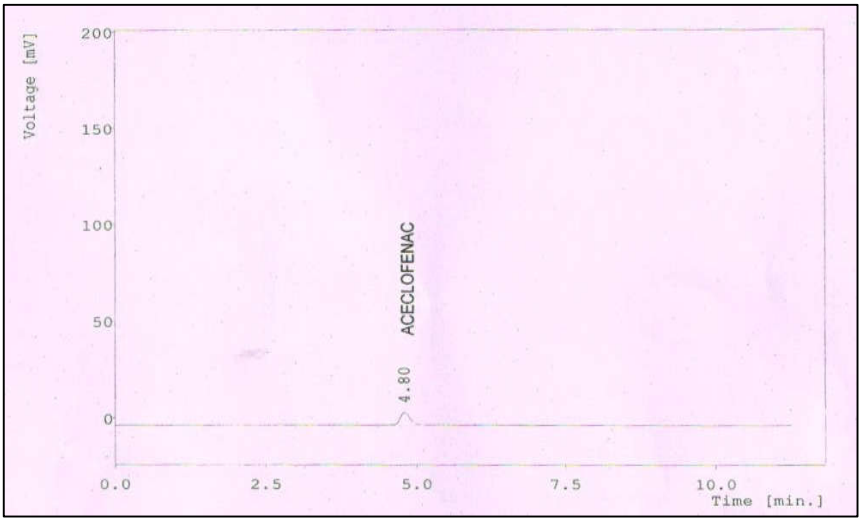

Fig 1: Chromatogram for Aceclofenac Standard

Table 1: Calculation for Aceclofenac Standard

Peak No	Retention Time	Area (mV.s)	Height (mV)	W05 (min.)	Area (%)	Height (%)
1	4.800	73.0352	6.3947	0.1800	100.0000	100.0000
-	Total	73.0352	6.3947			

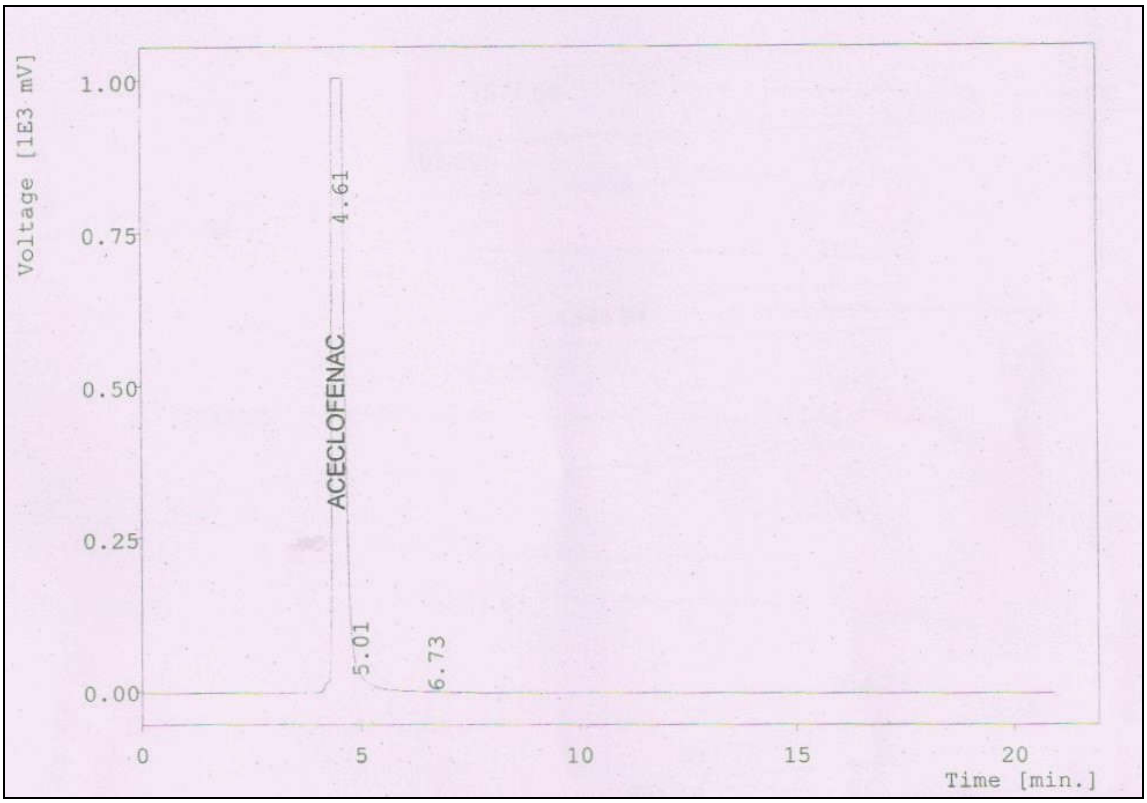

Fig 2: Chromatogram for Aceclofenac Sample

Table 2: Calculation for Aceclofenac Sample

Peak No	Retention Time	Area (mV.s)	Height (mV)	W05 (min.)	Area (%)	Height (%)
1	4.607	19859.1976	991.3155	0.3133	99.9985	99.9982
2	5.013	0.2534	0.0080	0.0133	0.0013	0.0008
3	6.727	0.0403	0.0095	0.0267	0.0002	0.0010
-	Total	19859.4913	991.3331			

3.2 FTIR -Concordance of sample with standard Aceclofenac

The FTIR analysis was carried out on Schimadzu IR Affinity-1 Spectrophotometer. FTIR Spectra were recorded in the wave number range between 1800 and 600 cm^{-1}, averaging 32 scans per sample using a nominal resolution of 4 cm^{-1}. The IR Solution software was used for data analysis and concordance/calibration curve plotting. The characteristic absorption peaks corresponding to Aceclofenac standard and sample were shown in Fig No 2. The absorption peaks for were 99.89% concordant.

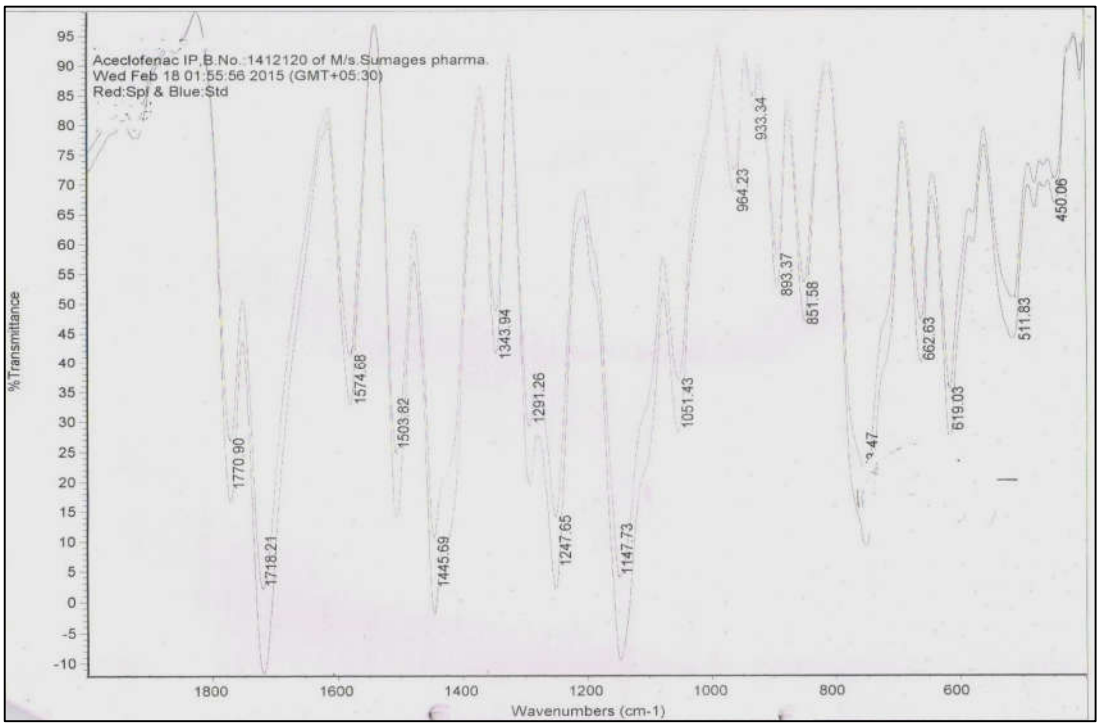

Fig 3: Concordance of Aceclofenac sample with standard-FTIR

4. CONCLUSION

The RP-HPLC and FTIR methods describe in the present paper was successfully employed in the analysis of Aceclofenac without any interference. As the reference drug 99.89% complies with the sample (both in terms of impurities as well as concordance), the proposed method is useful in the quality control analysis of Aceclofenac bulk drug and its solid dosage form manufacturing in order to build the overall quality of pharmaceutical dosage form.

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