

Comparative Study of Estimation of Clopidogrel Bisulphate from Commercial Sample By Uv – Visible Spectrophotometer and Hplc Method

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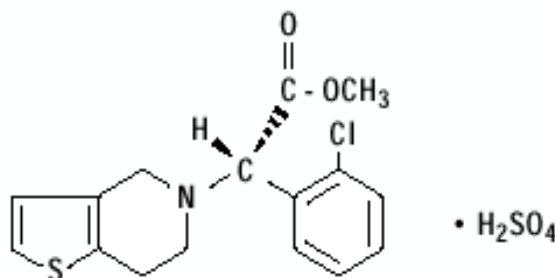
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ABSTRACT:- Clopidogrel bisulphate is an anti platelet drug. A Simple and accurate spectrophotometric and HPLC method was developed for determination of Clopidogrel bisulphate in tablets dosage form. The spectrophotometric method was developed by dissolving tablets in 1:1 methanol to make solution of 100 ppm giving absorbance at 220 nm. The experimental conditions were optimized and Beers law was obeyed over the applicable concentration range. The application of HPLC procedure depends on using a conventional reverse phase (C18) column along with mobile phase consisting of 1:1 Methanol. Both techniques were applied successfully for analysis of Clopidogrel bisulphate in three different commercially available tablets. From the results obtained for both procedures percentage purity was found out.

Keywords:- Clopidogrel bisulphate, spectrophotometric, HPLC method, Methanol

I. INTRODUCTION

Clopidogrel bisulphate is an antiplatelet drug. It is commonly encountered organic poison after Barbiturates. The lethal dose for an adult is very large but much more dangerous to children, who are fatally poisoned by it with distressing frequency. Its identification in fatal cases is easy because its low toxicity means that plenty will be recovered. CLOPIDOGREL BISULPHATE overdose causes hypertension which causes patients PCO₂ drop from normal limits. The body will try to compensate for this by excreting bicarbonates which result in increase in pH blood levels. If this is not corrected it will lead to metabolic alkalosis.(1) Literature survey showed number of methods of analysis for the detection of presence of salicylic acid and Acetyl salicylic acid in delayed release aspirin tablet by second derivative UV spectrophotometer.(2). Similarly Aspirin and paracetamol by UV spectrophotometer(3), There was successful investigation of salicylic acid by electrochemical oxidation on a glassy carbon electrode using cyclic voltammetry and differential pulse voltammetric (DPV) method (4). Aspirin, Paracetamol, Caffeine, and Chlorphenamine Using Multivariate Regression Methods by spectrophotometer, Acetylsalicylic acid, paracetamol, caffeine and phenobarbital by HPLC Method(5,6). But there was a need to develop rapid method for detection of presence of Aspirin quantitatively which require fewer chemicals and less manpower. The method was developed using different commercially available drug samples which are usually prescribed or bought over the counter. It is a white to off-white powder. It is practically insoluble in water. Following structure suggests total eight possible isomers. Out of them only one structure is an active antiplatelet drug.



CLOPIDOGREL BISULPHATE

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II. MATERIAL AND METHODS

- 1) UV-Visible spectrophotometer
- 2) HPLC - An Agilent 1200 liquid chromatograph equipped with a G 1312:BDegassar, G131SCQuartz form and DAD1315C detector was used for chromatographic measurements. The chromatograms were recorded and the peaks were quantities using automatic integrator.
- 3) COLUMN: Agilent ZORBAX SB-C18,
- 4) 1:1 methanol

2.1. **Preparation of mobile phase:** 0.1% Ortho phosphoric acid was prepared by dissolving 1ml of OPA in double distilled water and Acetonitrile in 30:70 proportion. It was ultrasonicated for 15 min.

2.2 Preparation of standard solutions

For spectrophotometric determination –Standard solution of Clopidogrel bisulphate was prepared of 100 ppm concentration. Using that standard solution a series of dilutions ranging from 2 ppm to 20 ppm was prepared.

For HPLC determination –

Standard solution of Clopidogrel bisulphate was prepared of 1000 ppm concentration. Using that standard solution a series of dilutions ranging from 40 ppm to 200 ppm was prepared.

2.3 Samples taken for analysis

TABLE NO.1

Name of Samples	Name of manufacturer	Batch number	Content per tablet
clopilet	Sun pharmaceutical industries	Eal0073	75 mg
clovix	Intas pharmaceuticals ltd	Kn129	75mg
clodrel	Unichem Laboratories ltd	Bcd11200	75mg

2.4 Preparation of Sample Solution For Analysis by Uv – Visible spectrophotometer

For Spectrophotometric determination twenty tablets were powdered in a mortar. Exact 100 mg of sample was taken in 25 ml volumetric flask and diluted with 1:1Methanol. The solution was left in ultrasonic bath for 5 min and then filtered through membrane filter . As per Table no. 2 according to Clopidogrel bisulphate present in given sample the corresponding volume was taken and volume made upto 25ml by 1:1 Methanol to get solution of 6 ppm of each sample.

TABLE NO.2

Sample	Wt. of sample tablet (Mg)	Clopidogrel bisulphate present in tablet (mg)	First dilution (ml)	Concentration of solution (ppm)	Volume required for final dilution (ml)	Final dilution (ml)	Final concentration (ppm)
clopilet	100	25.951	25	1038	0.12	25	6
clovix	100	25.337	25	1013	0.15	25	6
clodrel	100	24.916	25	996	0.15	25	6

2.5 Preparation of Sample Solution For Analysis by HPLC

For HPLC determination the contents of twenty tablets strip were powdered in a mortar. Exact 100 mg of sample was taken in 25 ml volumetric flask and diluted with 1:1Methanol. The solution was left in ultrasonic bath for 5 min and then filtered through .45um membrane filter . As per Table no. 3 according to Clopidogrel bisulphate present in given sample the corresponding volume was taken and volume made upto 25ml by 1:1 Methanol to get solution of 100 ppm of each sample.

TABLE NO.3

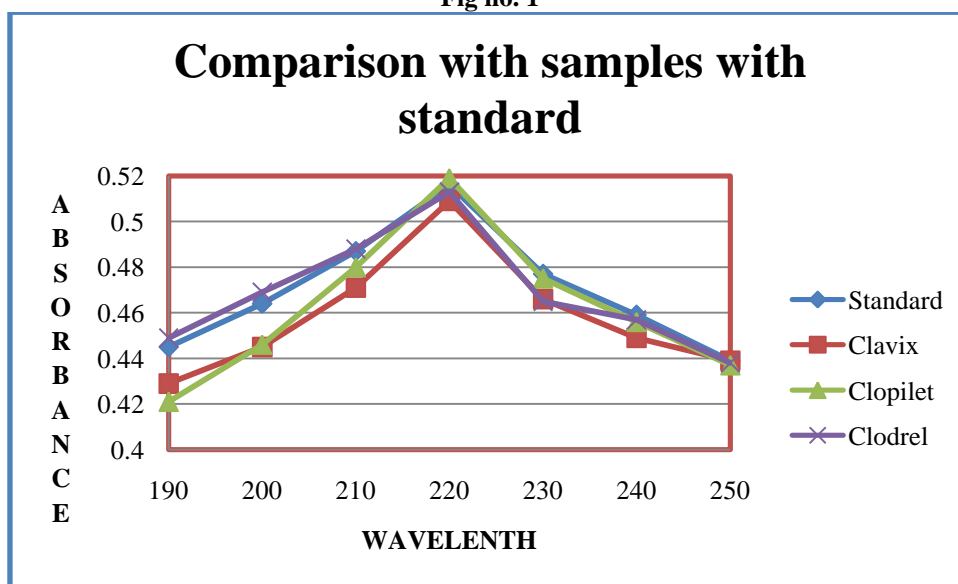
Sample	Wt. of sample tablet (Mg)	Clopidogrel bisulphate present in tablet (mg)	First dilution (ml)	Concentration of solution (ppm)	Volume required for final dilution (ml)	Final dilution (ml)	Final concentration (ppm)
clopilet	100	25.951	25	1038	2.40	25	100
clovix	100	25.337	25	1013	2.47	25	100
clodrel	100	24.916	25	996	2.51	25	100

III. RESULT AND DISCUSSION

3.1 Analysis by uv and visible spectrophotometer analysis.

For UV-visible spectrophotometer the λ_{max} value for standard solution of Clopidogrel bisulphate was found to be 220nm. Fig 1 shows the comparison of different sample solutions with standard sample. The calibration graph when plotted using different concentration of Clopidogrel bisulphate sample was found to be straight line passing through origin obeying Beer's law. Using this calibration graph values for samples were found out which are depicted in table no. 4. They were all found in range using their values percentage recovery was calculated which was found to be in range as shown in table no.4

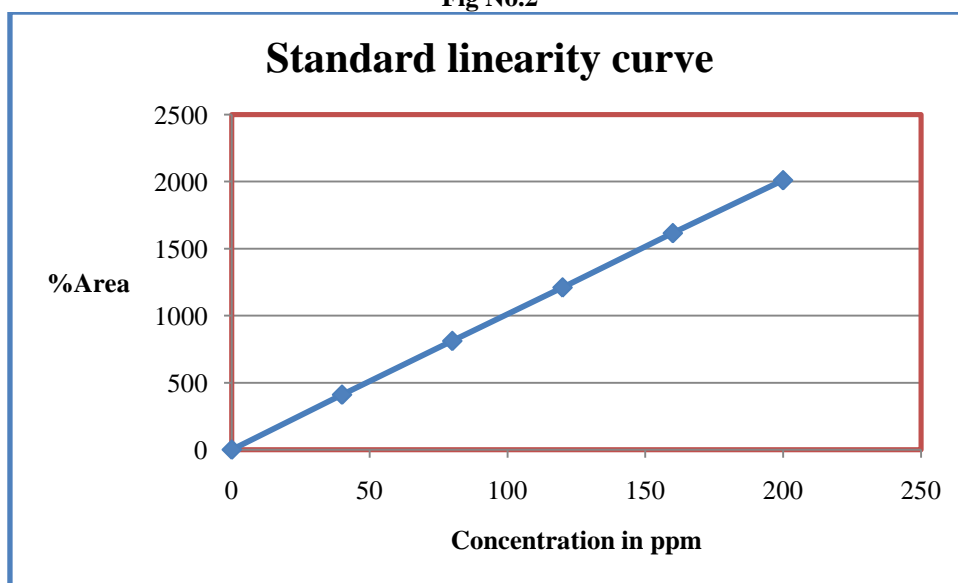
Fig no. 1



3.2 Analysis by HPLC method:

For HPLC measurements, the standard sample solutions of Clopidogrel bisulphate were prepared. Ranging from 40ppm to 200ppm. The standards were run and their %area was calculated. Using those values a standard linearity curve was plotted. This was observed that it follows Lambert Beer's law.

Fig No.2



After the system was set using standard sample the commercial samples were run and following results were obtained.

Table No. 3

Samples	% Area
Clavix	9835748
Clopilet	10146325
Clodrel	9969658

The following formula was used to calculate the percentage recovery of the sample by using both Uv and visible method and HPLC method.

$$\% \text{ Assay} = \frac{\text{OD of sample}}{\text{OD of std}} \times \frac{\text{std weight}}{\text{first dilution}} \times \frac{\text{vol of std solu required}}{\text{final dilu. Of std}} \times \frac{\text{dilu. of sampl}}{\text{CLOPIDOGREL BISULPHATE pres in samp}}$$

$$\% \text{ Recovery} = \frac{\% \text{ Assay}}{\text{Label claim on sample}} \times 100$$

IV. CONCLUSION

Despite the number of methods described by different researchers for analysis of CLOPIDOGREL BISULPHATE the proposed UV-VISIBLE Spectrophotometric method and HPLC method for determination of CLOPIDOGREL BISULPHATE in pharmaceutical samples is simple and rapid than other sophisticated instruments. All the samples analyzed were within the range as prescribed on tablet. These methods are very appropriate for routine analysis of active drugs in the laboratories. The procedures are easy to execute and require less sample handling than methods described in the literature. The following table gives the summary of result.

TABLE NO.5

Sample	% Recovery Of CLOPIDOGREL BISULPHATE By Uv-Visible Spectrophotometry Method	% Recovery Of CLOPIDOGREL BISULPHATE By HPLC Method
Clavix	95.01	94.30
Clopilet	99.22	99.64
Clodrel	99.73	99.56

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