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# A Simple Spectrophotometric Method for the Estimation of Prasugrel in Pharmaceutical Formulation

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**ABSTRACT**

A simple, sensitive, rapid and accurate spectrophotometric method has been developed for the estimation of prasugrel in bulk and pharmaceutical dosage forms. The method is based on the reaction of prasugrel with 2, 3-dichloro-5, 6-dicyano-1, 4-benzoquinone (DDQ) to form a red colored charge-transfer complex. The red colored solution is used to determine the prasugrel spectrophotometrically.

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## INTRODUCTION

Prasugrel is scientifically termed as [5-[2-cyclopropyl-1-(2-fluorophenyl)-2-oxoethyl]-6,7-dihydro-4H-thieno[3,2-c]pyridin-2-yl] acetate. Prasugrel is a novel platelet inhibitor approved for the reduction of thrombotic cardiovascular events in patients with acute coronary syndrome. Prasugrel produces inhibition of platelet aggregation by binding to receptors for ADP on the surface of platelets. Several analytical methods have been reported for assay of prasugrel including spectrophotometric method [1-9], RP-HPLC method [10-13], HPLC method [14-16], Chromato method [17], Electrode method [18,19], UPLC method [20], Pellet method [21].Molecular Formula: $C_{20}H_{20}FNO_3S$, Molecular Weight: 373.4 g/mol, Prasugrel is having the following Fig.1:

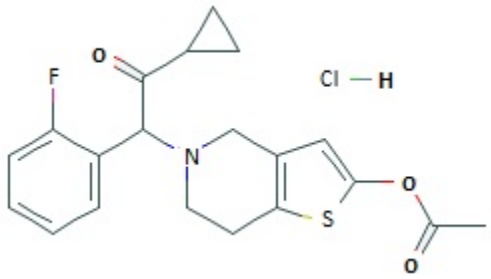


**Figure No. 1: Structure of Prasugrel**

The method is based on the reaction of prasugrel with 2, 3-dichloro-5, 6-dicyano-1, 4-benzquinone (DDQ) to form an orange red colour charge-transfer complex. The orange red colour solution is used to determine the prasugrel spectrophotometrically.

## EXPERIMENTAL

## MATERIALS AND METHOD

### Instrument:

All measurement were done on Milton Roy 1001spectrophotometer by using 10 mm matched quartz cuvettes.

**Preparation of reagents and solutions:**

**DDQ (100µg/ml):**

DDQ (2,3-dichloro 5,6-dicyano-p-benzoquinone) (Loba Chem., India) is prepared by dissolving 80 mg of DDQ in 100 ml of methanol and 5 ml of this stock solution is dissolved in 40 ml methanol.

**Spectrum of Prasugrel treated with DDQ:** The wavelength of maximum absorbance of the prasugrel drug treated with DDQ solution is ascertained by the following procedure. 1ml of prasugrel solution (100µg/mL) is transferred into a standard flask. To this solution 1.0 ml of DDQ reagent is added to form an orange red colour solution. The final volume is brought to 10 ml with methanol. The resultant solution is well mixed and allowed to stand for 5 min for complete the reaction. The absorbance of the orange red colour solution is measured in the wavelength range of 300 to 550 nm, against the reagent blank. The spectrum is given in fig.2.

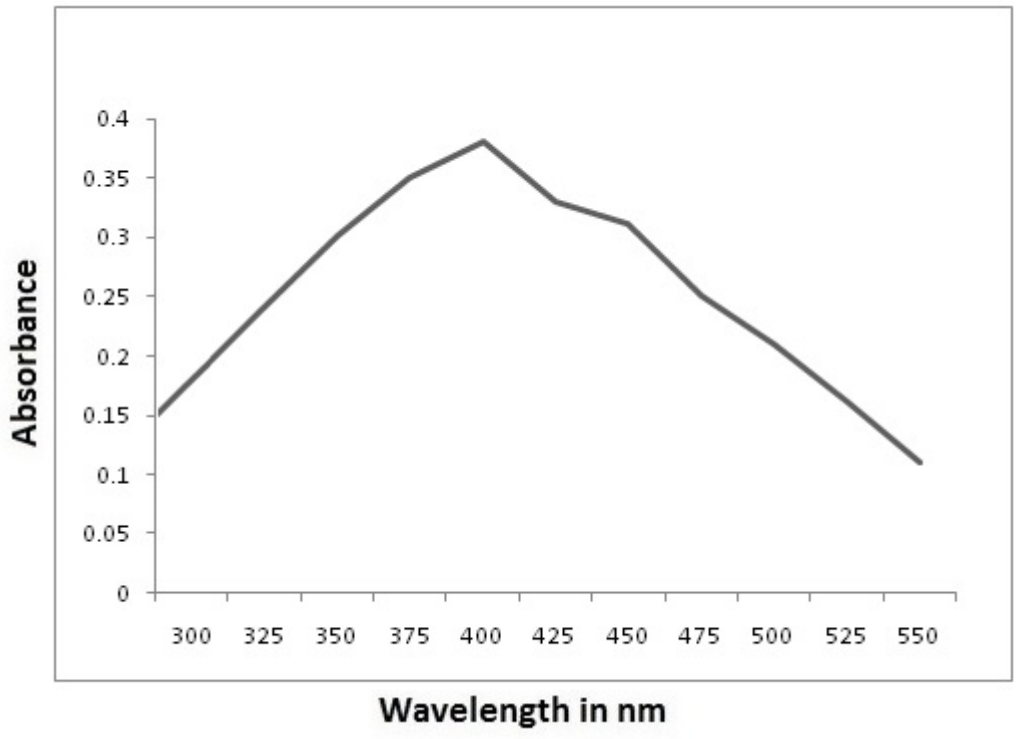


**Figure No. 2: Absorption spectrum of Prasugrel**

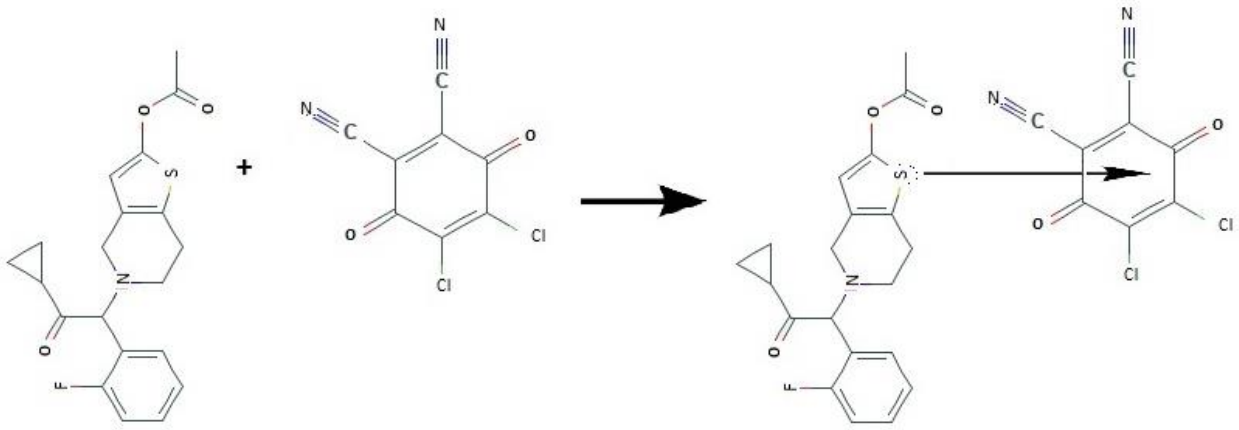

**Figure No. 3: Reaction scheme of prasugrel with DDQ to form charge-transfer complex**

From fig.2, it is clear that the prasugrel drug treated with DDQ solution has maximum absorbance at 393 nm. Hence, all further studies are made at 393 nm.

The optimal conditions for the determination of prasugrel are arrived at by the following steps.

**Effect of concentration of** DDQ **solution on the absorbance of charge transfer complex is studied by the following procedure:**

In a series of standard flask, 1.0 ml of prasugrel are taken and varying amounts of DDQ solution are added. The contents are made up to the mark with methanol. Reaction mixture was shaken gently for 5 min and allowed to stand for 5 min to complete the reaction. The absorbance of the resultant solutions is measured at 393 nm and the data are presented in table.1.

**Table No. 1: Effect of concentration of DDQ solution**

| Volume of DDQ solution (ml) | Absorbance at 393 nm. |
|---|---|
| 0.5 | 0.375 |
| 1.0 | 0.435 |
| 1.5 | 0.428 |
| 2.0 | 0.429 |

The data in table 1 indicate that 1.0 ml of DDQ is necessary for a chieving maximum absorbance and hence maintained throughout the experimental studies.

**Assay Procedure:**

Various aliquots of the standard prasugrel solution ranging from 0.2-1.0 ml are transferred into a series of standard flasks. To each flask, 1.0 ml of DDQ solution is added to produce an orange red color. The final volume is brought to 10 ml with methanol. The reaction mixture in each flask is well shaken and allowed to stand for 5 min to complete the reaction. The absorbance of the orange red colour solution is measured at 393 nm, against the reagent blank prepared in similar manner omitting drug solution. Calibration graph is obtained by plotting absorbance values against the concentration of prasugrel solution. The calibration curve is found to be linear over a concentration range of 20 to 100 µg/mL of prasugrel. The amount of prasugrel present in the sample is read from the calibration graph. The results are presented in fig.4.

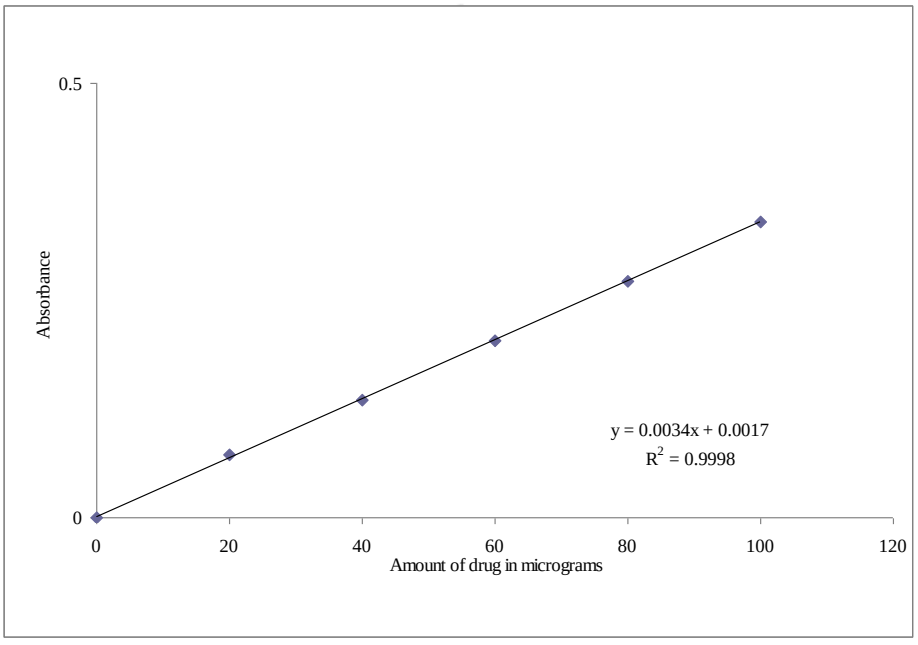


**Figure No. 4: Calibration curve of Prasugrel**

**Assay of Prasugrel in bulk samples:**

50 mg of pure prasugrel is dissolved in methanol and the volume is made up to 50 ml with methanol. Further dilution is made as described in the preparation of standard solution of prasugrel. Further analysis is carried out as per procedure described above and results are

summarized in Table.3. The amount of drug present in the sample is estimated from calibration graph.

## RESULTS AND DISCUSSION

In this method, the drug react with DDQ solution to form an orange red charge complex. The orange red coloured charge complex solution formed is measured at 393 nm against reagent blank. The amount of drug read from calibration curve. The calibration curve is linear over the range of 20-100 µg/mL of prasugrel. The optical characteristics of the proposed method such as absorption maxima, Beer´s law limits, molar absorptivity and Sandell´s sensitivity are presented in Table 2. The molar absorptivity and Sandell´s sensitivity values show sensitivity of the method. The regression analysis using method of least squares was made for the slope (b), intercept (a) and correlation (r) obtained from different concentrations and results are summarized in Table 2. The value of correlation coefficient (r) was 0.999, which indicated the good linearity of calibration lines.

**Table No. 2: Optical characteristics of the proposed methods**

| **parameters** | **Proposed method** |
|---|---|
| λmax (nm) | 393 |
| Beer's law limit (µg/ml) | 20-100 |
| Molar absorptivity (l mole-$^{1}$ cm-$^{1}$) | 4.205x10$^{3}$ |
| Sandell's sensitivity (µg cm-$^{2}$ / 0.001 absorbance unit) | 0.08879 |
| Regression equation (Y = a + bx) | Y=0.0034X+0.0017 |
| Slope (b) | 0.0044 |
| Intercept (a) | 0.0017 |
| correlation coefficient (r) | 0.9995 |

*Y = a+bx, where Y is the absorbance and X concentration in 50 µg/ml

**Table No. 3: Assay of Prasugrel in tablet formulations**

| Tablets | Labeled amount(mg) | *Amount found (mg)±S.D* | % label claim | %RSD* | *t value |
|---|---|---|---|---|---|
| Tablet 1 | 10 | 10.02±0.33 | 100.2 | 0.4181 | 0.1070 |
| Tablet 2 | 10 | 10.06±0.38 | 10.6 | 0.4817 | 0.8130 |
| Tablet 3 | 10 | 9.98±0.51 | 99.84 | 0.6460 | 0.0865 |

*Average of five determination based on label claim

## CONCLUSION

The percent relative standard deviation calculated from the five measurements of flupirtine shown in Table 3. The % RSD is less than 2, which indicates that the method has good reproducibility. The values of standard deviation values are low, indicates high accuracy and reproducibility of the method. The 't' calculated values are compares well with the theoretical value of 2.78 thereby indicating that the precision of the method.

The proposed method is found to be simple, precise, accurate and time saving, reproducible and can be conveniently adopted for routine analysis of estimation of flupirtine in bulk drugs samples.

## REFERENCES

1. A. Ashok Kumar, A. Anil Kumar and D. GowriSankar, Development, Estimation and Validation of Prasugrel in bulk and in its Pharmaceutical Formulation by UV Vis Spectroscopic Method, An International Journal of Advances In Pharmaceutical Sciences, 2(1), 37-39, 2011.
2. Ranjith Kumar Rajendiran, Vinoth Kumar Sekar, Bharatha Devi Namadevan, Jai Kumar Annamalai, UV-Spectrophotometric and RP-HPLC Method for the Estimation of Prasugrel Hydrochloride in bulk and Tablet Formulation, International Journal of Pharmacy and Pharmaceutical Sciences, 6(1), 220-225, 2014.
3. Camila Rigobello, Amanda Thomas Barden and Martin Steppe, Development and validation of the discriminating method of prasugrel dissolution in tablets using ultraviolet detection, Acta Scientiarum. Health Sciences, 36(2), 235-241, 2014.
4. Syed SadathKabir, Abdul Naveed, Syed Zia UlQuasim, Vijay Krishna Nandipati, Spectrophotometric Method Development and its Validation for Estimation of Prasugrel HCl in Bulk and Pharmaceutical Formulations, Research J. Pharm. and Tech, 6(6), 641-644, 2013.
5. Shahabuddin N. Alvi, Mehul N. Patel, Prakash B. Kathiriya, Bhavna A. Patel, and Shraddha J. Parmar, Simultaneous Determination of Prasugrel and Aspirin by Second Order and Ratio First Order Derivative Ultraviolet Spectrophotometry, 2013(1), 1-7, 1013.
6. M Chennaiah, Extractive Spectrophotometric Methods for determination of Prasugrel Hydrochloride in Pharmaceutical Formulations using some Acidic Triphenylmethane Dyes, Asian J. Research Chem. 9(8), 373-377, 2016.

7. Camila Rigobello, Amanda Thomas Barden and Martin Steppe Faculdade de Farmácia, Development and validation of the discriminating method of prasugrel dissolution in tablets using ultraviolet detection, Acta Scientiarum. Health Sciences Maringá, 36(2), 235-241, 2014.
8. Satya Prasad B and Jayakumar, Quantification of Anticoagulants Dabigatran, Rivaroxaban and Prasugrel by Chromatographic and Spectrometric Techniques, AJPCR, 12(4),1-8, 2019.
9. Harshini, B.; Alekhya, S. V. R.; Manasa, G.; Prakash, K. V., Extractive spectrophotometric estimation of Prasugrel in pharmaceutical formulation, Research Journal of Pharmaceutical, Biological and Chemical Sciences, 2(3), 431-437, 2010.
10. P. Vivek Sagar, T. Samidha, M. Vamshi Krishna and S. Shobha Rani, A Validated RP-HPLC Method for Simultaneous Estimation of Aspirin and Prasugrel in Tablet Dosage Form, IJPSR, 5(11), 4858-4864, 2014.
11. Deepak Kumar Jain, Nilesh Jain, Jitendra Verma, RP-HPLC Method for Simultaneous Estimation of Aspirin and Prasugrel in Binary Combination, International Journal of Pharmaceutical Sciences and Drug Research, 4(3), 218-221, 2012.
12. M. Naresh Chandra Reddy, K. B. Chandra Sekhar, A. Kavitha, E. Sasikiran Goud, Development and Validation of a RP-HPLC Method for Related Substances of Prasugrel for 5 and 10 mg Tablets, Int J Pharm Sci, 6(1), 90-94, 2013.
13. Viralkumar J. Modi and Prashant L. Pingale, Development and Validation of Analytical Method for Estimation of Prasugrel Hydrochloride in Bulk and Pharmaceutical Formulations, 3(4), 292-298, 2012.
14. Venugopal Sriram, Kalpana Sriram, Jagadishbabu Angirekula, Upendra M. Tripathi and Devanna Nayakanti, Development And Validation of Reverse Phase HPLC Method For The Determination of Impurities In Prasugrel Hydrochloride, International Journal of Pharm Tech Research, 4(4), 1407-1416, 2012.
15. G. Usha Rani, B. Chandrasekhar and N. Devanna, Analytical method development and validation of prasugrel in bulk and its pharmaceutical formulation using HPLC, Journal of Applied Pharmaceutical Science, 1(7), 172-175, 2011.
16. Shital M. Patel, C. N. Patel, and V. B. Patel, Stability-indicating HPLC Method for Simultaneous Determination of Aspirin and Prasugrel, Indian J Pharm Sci., 75(4), 413–419, 2013.
17. Samer Housheh, Saleh Trefi, Mohammad Haroun, M. Fawaz Chehna, Analysis of Prasugrel by Chromatography – Review, Int. J. Pharm. Sci. Rev. Res., 28(2), 180-186 2014.
18. Mohammad Haroun, Dania Nashed, Amir Alhaj Sakur, New electrochemical methods for the determination of Prasugrel using drug selective membranes, International Journal of Academic Scientific Research, 5(3), 30-36, 2017.
19. Amir Alhaj Sakur, Dania Nashed, Mohammad Haroun, Imad Noureldin, Determination of Prasugrel Hydrochloride in Bulk and Pharmaceutical Formulation Using New Ion Selective Electrodes, Research J. Pharm. and Tech., 11(2), 631-636, 2018.
20. Kapendra Sahu, C. Karthikeyan, N. S. Hari Narayana Moorthy & Piyush Trivedi, Comparative Study of Forced Degradation Behaviour of Prasugrel by UPLC and Development of Validated Stability Induced Assay Method, Journal of Liquid Chromatography & Related Technologies, 34(17), 1870-1884, 2011.
21. Navjot Kanwar, Rakesh Kumar, V.R. Sinha, Preparation and Evaluation of Multi-Particulate System (Pellets) of Prasugrel Hydrochloride, OPS Journal, 2(1), 74-80, 2015.

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