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UV spectroscopy and HPLC method Development for the Estimation of Aspirin and Ticgrelor in Marketed formulation

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Abstract

Proposed analytical methods for the estimation of Aspirin and Ticagrelor in marketed formulations have been taken for this research work. These methods stand out for their simplicity, rapidity, accuracy, and reproducibility, making them highly suitable for practical applications in pharmaceutical analysis. The linear range of 5-25 µg/ml for both Aspirin and Ticagrelor, with correlation coefficients close to one, demonstrates the robustness and reliability of the proposed methods. The validation and reliability of these methods were rigorously assessed through recovery studies, which yielded satisfactory results, ranging from 99.102% to 99.56% for Aspirin and 99.48% to 98.54% for Ticagrelor at various added standard concentrations (80%, 100%, and 120%). This indicates that the methods are capable of providing accurate and precise results in the analysis of these pharmaceutical compounds.

Precision was evaluated through various parameters, including repeatability, intermediate precision, and reproducibility, all of which demonstrated the consistent and reliable performance of the methods. The robustness of the developed method, tested by introducing deliberate variations in solvent, further confirmed the versatility and adaptability of the methods.

Keywords: Aspirin, Ticagrelor, Precision, Reproducibility, Accuracy

1. Introduction

A crucial aspect of the pharmaceutical industry revolves around the ability to generate and deliver data that is precise, reliable, and promptly available throughout the process of exploration, formulation, and production of drugs. Analytical data is utilized for the evaluation of potential pharmaceutical candidates, facilitation of drug synthesis development, provision of support for formulation studies, monitoring of bulk medicine and product stability, and execution of release testing on finalized products. The significance of high-quality analytical data in ensuring the success of a drug development program cannot be emphasized enough [1-3].

When more people, laboratories, and equipment are used to execute a process, complications increase. Once the method is disseminated to other laboratories or employed for official product testing, where methods are presented to regulatory agencies and modifications may necessitate formal approval prior to implementation for official testing, the developer relinquishes the capacity to make minor alterations to the method. Performing proper validation experiments during method development is the best way to minimise method issues [4].

For modern analytical laboratories, the use of advanced analytical tools and determination is almost a matter of course. The ever-increasing power and scope of instrument automation has led to a significant expansion of

knowledge. In addition, all manual procedures in the field of analytical investigations were gradually replaced by instrumental approaches. Physical and chemical analysis is the most common types of analytical techniques. Physical analysis encompasses the assessment of various parameters such as particle size, dimensions, and viscosity of solid dosage forms, among other factors [5]. Chemical analysis can be classified into three primary categories:

1. Qualitative analysis refers to the process of identifying the constituent element(s) present within a given sample.
2. Quantitative analysis is an analytical method used to determine the absolute quantity of a specific species within a given sample.
3. The process of evaluating the behavior and performance of a structure under various loads and conditions, typically using mathematical models and engineering principles. Molecular analysis involves determining the spatial configuration of atoms within a molecule and identifying the presence or location of specific organic functional groups in a given compound.

2. Materials and Instruments

Materials

Chemicals and solvents

- Acetonitrile - HPLC grade (Qualigens)
- Methanol - Qualigens
- ammonium carbonate - Merck

Spectrophotometric Method Development for Aspirin and Ticagrelor

Preparation of standard stock solution

Precisely measured 10 milligrams of aspirin and ticagrelor were individually placed into separate 50 milliliter volumetric flasks. The substances were dissolved in 10 milliliters of acetonitrile, and then the volume was adjusted to 50 milliliters using acetonitrile. The mixture was then agitated using a vortex mixer to ensure complete dissolution of the drugs. Set it aside for a few minutes. The concentration of aspirin and ticagrelor was 200µg/ml. The stock with the symbol "A" A volume of 5 milliliters of solution was extracted from stock-A of aspirin and transferred into a 10 milliliter volumetric flask. It was then diluted with diluent (Acetonitrile) up to a total volume of 10 milliliters, resulting in a concentration of 100 micrograms per milliliter (Stock-B) [6-8].

Determination of Wavelength Maxima (λ_{max})

The 10µg/ml solution was prepared by withdrawing 10 ml of solution from stock B solution and further diluted with acetonitrile to get concentration. This solution was then scanned at a wavelength of 200-400 nm against the blank. The lambda max was found to be at 260 and 282nm. Hence this is considered as absorption maxima which are used for preparation of calibration curve [9-11].

Method Validation

The method underwent validation in accordance with the guidelines set by the International Council for Harmonisation (ICH) and the Food and Drug Administration (FDA). The validation process assessed various parameters, including specificity, linearity, range, accuracy, precision, sensitivity (limit of quantification and limit of detection), and robustness.

Linearity

The linearity of an analytical procedure pertains to its ability, within a defined range, to generate test outcomes that exhibit a direct correlation with the quantity of analyte contained in the sample. The calibration plot was generated by conducting an analysis on a set of five distinct concentrations, spanning from 5 to 25 µg/ml. Three measurements were taken for each concentration, and the mean area was computed. The user has provided the mathematical equation for the regression model and the value of the correlation coefficient, which measures the strength of the

relationship between variables. Additionally, the user has included a visual representation of the standard calibration curve for the drug. The response ratio, also known as the response factor, was calculated by dividing the area under the curve (AUC) by its corresponding concentration value [12-14].

Specificity

The method was conducted with a high level of specificity to definitively determine the presence of the analyte, including any impurities, degradation products, and matrix components that could potentially be present.The separation between the analyte peak and the nearest peak. Each analyte was individually injected and their respective retention times were recorded. The conventional operational method involves injecting a solution that comprises a blend of the component under analysis. Each peak of the analyte is subsequently assessed to determine its resolution [15].

Accuracy

Validation experiments were conducted to assess the precision and reliability of the developed methodology. The preanalyzed sample solution was supplemented with a known concentration of standard drug (80%, 100%, and 120%) and subsequently subjected to recovery analysis.

Robustness

In accordance with the International Council for Harmonisation (ICH) guidelines, intentional and minor adjustments were made to the concentration of the mobile phase in order to assess the method's ability to remain unaffected. The mobile phase ratio was modified from 20mM Phosphate Buffer: acetonitrile (80:20 %v/v) to (85:15 % v/v) [16-17].

Method Development by RP-HPLC

The RP-HPLC technique was employed to determine the quantities of aspirin and ticagrelor in both bulk and tablet forms. Isocratic elution was performed using a mobile phase consisting of 20 mM KH_2PO_4 and acetonitrile (pH 3.0) in a volumetric ratio of 20:80. The stationary phase utilized was a Thermo C-18 column with dimensions of 4.6 x 250mm and a particle size of 5µ. The resulting chromatogram was recorded at a wavelength of 275nm. The developed method underwent validation by employing multiple parameters [18].

System suitability

System suitability is a critical aspect of any analytical method and serves as a fundamental step in ensuring the reliability and validity of the results obtained from an analytical system, such as high-performance liquid chromatography (HPLC). It involves a series of tests and criteria to assess the performance of the system under specific operating conditions. The objective of conducting system suitability tests is to confirm that the analytical system is functioning properly and can provide accurate and precise results [19].

The system suitability parameter was conducted to validate the functionality of the analytical system and its ability to provide accurate and precise results. Six replicates of the reference standard solution, each containing 10 µg/ml of aspirin and ticagrelor, were injected separately into the HPLC system. These replicates serve as a representation of the samples to be analyzed.

Linearity

Linearity is a fundamental attribute of an analytical technique, which is evaluated to ascertain the technique's capacity to produce test outcomes that exhibit a direct correlation with the concentration of the substance being analyzed in a sample within a specified range. In the realm of high-performance liquid chromatography (HPLC) or similar analytical techniques, it is imperative to conduct a linearity assessment. This assessment is crucial in verifying the method's ability to precisely measure the concentration of analytes within a designated range in a given sample [20].

The linearity of the method is assessed by examining the calibration curve. Linearity Range is the concentration range over which the method is expected to provide accurate and proportional results. It is determined by the data

collected from the standard solutions. Correlation Coefficient (R^2) with high value close to 1 indicates good linearity. It quantifies the strength and direction of the relationship between concentration and detector response. A residuals plot is examined to assess whether the differences between the observed and predicted values are random and evenly distributed. A well-behaved residuals plot is indicative of good linearity. The acceptance criteria for linearity are typically based on regulatory guidelines and the specific needs of the analytical method. Generally, an R^2 value greater than 0.99 and a well-behaved residuals plot are indicators of good linearity.

The linearity of the analytical method was assessed to determine its capacity to produce test results that exhibit a proportional relationship to the concentration of the analyte in the sample within a specified range. Various concentrations of standard solutions were prepared and introduced into the High-Performance Liquid Chromatography (HPLC) system, and the resulting chromatogram was recorded.

Specificity

The specificity of the analytical method is a crucial parameter to assess its ability to selectively identify and quantify the analytes of interest, in this case, aspirin and ticagrelor, in the presence of potential interferences. In this study, the specificity of the method was rigorously examined. The results indicate that the peaks corresponding to the diluent, mobile phase, and excipients present in the tablet formulations did not interfere with the standard peaks of aspirin and ticagrelor. This outcome underscores the method's capacity to distinguish and accurately measure the analytes of interest in complex sample matrices, thereby ensuring the reliability and precision of the analysis. The demonstrated specificity is a vital aspect of method validation, and quality control applications, where the presence of potential interferences can impact the accuracy of analytical results and the safety of the final product [20].

Accuracy

To establish the validity and reliability of the proposed analytical methods, an accuracy assessment was conducted through recovery studies. This involved the addition of standard analytes at three different levels (80%, 100%, and 120%) to test samples, and the analysis was replicated three times at each concentration level. The accuracy of the method was determined based on the observed recovery values. The results showed that the recovery percentages were in close proximity to 100%, indicating that the method yielded results very close to the true values. Additionally, the standard deviation (SD) and the relative standard deviation (% RSD) associated with these recovery values were both less than 2, which is a widely accepted criterion for accuracy. This outcome affirms that the methods are highly accurate in quantifying the analytes, instilling confidence in their suitability for quantitative analysis in the field of analytical chemistry or other relevant applications. Accuracy is a fundamental aspect of method validation, and these findings underscore the precision and reliability of the proposed analytical methods [20].

Precision

Precision, an essential parameter in method validation, was assessed through two key components: repeatability and intermediate precision. Repeatability was evaluated to understand the precision of the method under the same operating conditions and over a short interval of time. The results from the repeatability study provide insight into the method's reliability and consistency when applied repeatedly.

The intermediate precision assesses the variation within the laboratory on different days and the variation introduced by different analysts. By analyzing intermediate precision, the method's robustness and ability to produce consistent results regardless of minor changes in laboratory conditions or operators are evaluated.

In both cases, precision is considered acceptable when the standard deviation (SD) and the relative standard deviation (% RSD) associated with the test results are less than 2. A % RSD value below 2% is indicative of excellent precision, signifying that the method produces highly reproducible and consistent results, essential for its reliability in various analytical applications. The precision assessment contributes to the overall method validation, instilling confidence in the method's ability to consistently and accurately measure the analytes of interest [19-20].

Robustness

The robustness of the developed method for the analysis of Aspirin and Ticagrelor was assessed by deliberately introducing variations in the solvent used. This robustness testing involves deliberately altering the solvent composition or conditions to evaluate the method's ability to withstand such changes without compromising the accuracy and reliability of the analytical results. Robustness studies are a critical aspect of method validation, as they demonstrate the method's resilience to minor deviations in experimental parameters. The method's ability to maintain its performance even when subjected to these controlled variations in solvent conditions ensures its suitability for application in different laboratory settings and under slightly different circumstances. It underscores the method's adaptability and its capacity to consistently deliver accurate results in various analysis [18-20].

Limit of Detection (LOD) and Limit of Quantification (LOQ)

The LOD and LOQ are critical parameters in HPLC method development. They determine the lowest concentrations of analytes that can be reliably detected (LOD) and quantified (LOQ) with acceptable accuracy and precision. These parameters are essential in assessing the sensitivity and performance of the HPLC method for the analysis of Aspirin and Ticagrelor.

In HPLC method development for Aspirin and Ticagrelor, LOD and LOQ are determined experimentally. Typically, a series of solutions with decreasing analyte concentrations is analyzed, and the signal responses are recorded. The LOD is then calculated based on the S/N ratio where the signal is just discernible above the noise, and the LOQ is determined at a higher S/N ratio where accurate quantification can be achieved [18-20].

Limit of Detection (LOD)

The LOD is the lowest concentration of an analyte that can be detected but not necessarily quantified. It is typically associated with a signal-to-noise ratio (S/N) of 3:1 or 2:1, indicating that the analyte's signal is three or two times higher than the noise level. LOD is essential for identifying the presence of an analyte in a sample, particularly in trace analysis

Limit of Quantification (LOQ)

The LOQ is the lowest concentration of an analyte that can be quantified with acceptable precision and accuracy. It is associated with a higher S/N ratio, often 10:1. LOQ is used to determine the minimum concentration at which the analyte can be reliably quantified. It is crucial for accurate quantification in routine analysis.

3. Results and Discussion

Result of Method Developed by UV spectrophotometry

Wevelength Maxima

The Wevelength Maxima for aspirin and ticagrelor were found to be at 260nm and 282nm. Results were found to be very closed to the reportd method (Fig 5.1 and 5.2).

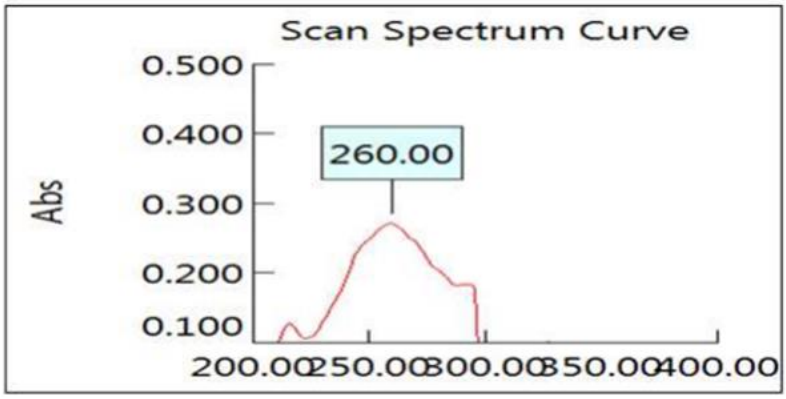

Figure 1: λ_{max} of Aspirin

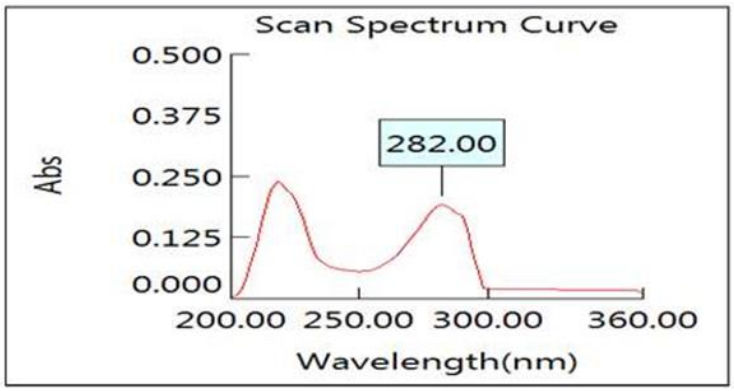

Figure 2: λ_{max} of Ticagrelor

Preparation of calibration curve

Calibration curves of standard drugs and marketed tablets were prepared at λ_{max} of 260 and 282nm by using UV-Visible spectrophotometer.

Table 1: Calibration Data for Standard Aspirin at 260 nm

S. No.	Concentration (µg/mL)	Absorbance
1	5	0.0712
2	10	0.1378
3	15	0.2131
4	20	0.2815
5	25	0.3482
6	30	0.4226

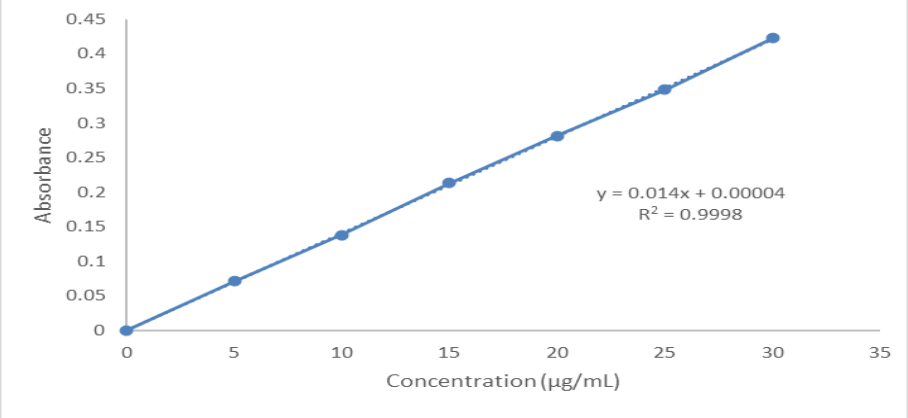

Figure 3: Calibration Data for Standard Aspirin at 260 nm

Table 2: Calibration Data for Standard Ticagrelor at 282 nm

S. No.	Concentration (µg/mL)	Absorbance
1	5	0.1269
2	10	0.2440
3	15	0.3574
4	20	0.4702
5	25	0.5986
6	30	0.7178

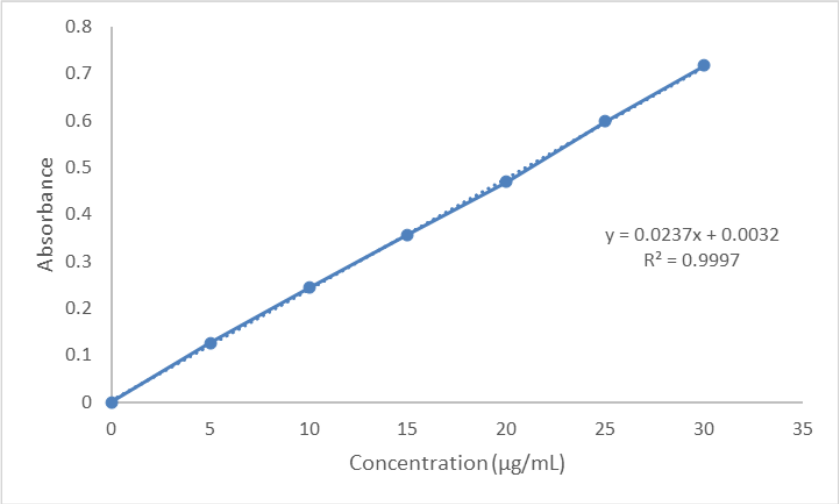

Figure 4: Calibration Data for Standard Ticagrelor at 282 nm

Table 3: Calibration Data for Aspirin and Ticagrelor Mixture

S. No.	Concentration (µg/mL)	Absorbance Aspirin	Absorbance Ticagrelor
1	5	0.0704	0.1235
2	10	0.1404	0.2458
3	15	0.2102	0.3569
4	20	0.2797	0.4744
5	25	0.3490	0.5951
6	30	0.4247	0.7149

Validation of the Spectrophotometric method

Linearity and Range

The linearity was found to be found to be of 5-30µg/mL for Aspirin and Ticagrelor. Correlation co-efficient for calibration curve of Aspirin and Ticagrelor was found to be 0.9998 and 0.9997 respectively. The regression line equation for Aspirin and Ticagrelo are as following,

$$Y \text{ (Aspirin)} = 0.014x + 0.00004$$

$$Y \text{ (Ticagrelo)} = 0.0237\ x + 0.0032$$

Correlation coefficients for both the drugs were near to one, indicate linearity of method within the given range.

Specificity

In order to verify the absence of interferences of the excipients on the analysis of Aspirin and Ticagrelor in Torplat A tablets, we carried out the analysis of a sample prepared with all the excipients present in the tablets, but without the drug (in house mixture of the excipients). Absorption spectra did not show any potential interference of the tablet excipients at 260nm and 282nm.

Precision

Precision is a critical aspect of method validation, and it assesses the ability of the method to produce consistent and reproducible results under different conditions and across multiple runs. The precision parameters provided in the table 4 demonstrate the ability of the Simultaneous Equation Method to produce reliable and consistent results for both Aspirin and Ticagrelor under various conditions. Low %R.S.D. values suggest that the method is precise, with little variability in the results, which is essential for analytical methods used in quality control and research settings.

Table 4: Precision of Drugs

Parameter		Simultaneous Equation Method	
		Aspirin	**Ticagrelor**
Precision (%R.S.D.)	Repeatability	0.033	0.043
	Day to Day	0.050	0.038
	Analyst to Analyst	0.033	0.150
	Reproducibility	0.071	0.039

Repeatability, also known as intra-day precision, evaluates the method's precision when the same analyst analyzes the same samples on the same day, typically under the same operating conditions. The %R.S.D. (Relative Standard Deviation) provides a measure of the variability in the results. For Aspirin, the repeatability %R.S.D. is 0.033, indicating very low variability, while for Ticagrelor, it's slightly higher at 0.043.

Day-to-day precision assesses the method's repeatability over different days. It measures how consistent the results are when the same analyst analyzes the same samples on different days. For Aspirin, the day-to-day %R.S.D. is 0.050, and for Ticagrelor, it's 0.038, indicating that the method maintains good consistency over time.

Analyst-to-analyst precision evaluates the variation in results when different analysts perform the analysis on the same samples. It measures the method's robustness against different operators. In this case, for Aspirin, the %R.S.D. is 0.033, indicating good consistency, while for Ticagrelor, it's 0.150, suggesting slightly higher variability between analysts.

Reproducibility assesses the method's precision across different laboratories or with different HPLC instruments. It gauges how consistent the results are when the method is applied under varying conditions. For Aspirin, the reproducibility %R.S.D. is 0.071, indicating a reasonable level of consistency, while for Ticagrelor, it's 0.039, indicating good reproducibility.

Accuracy

The accuracy of the method was evaluated through recovery experiments. Recovery experiments were conducted by introducing a predetermined quantity into the tablet. The recovery process was conducted at three different levels, specifically 80%, 100%, and 120% of the standard concentration for both Aspirin and Ticagrelor. These details can be found in Table 5-6. The recovery samples were prepared according to the specified procedure. A total of three samples were prepared for each recovery level. The solutions were subsequently examined, and the percentage recoveries were determined based on the calibration curve. The recovery values were determined as follows:

$$Accuracy = \frac{Amount\ of\ Sample\ Conc.Found\ -\ Amount\ of\ Test\ Conc\ taken}{Amount\ of\ Standard\ Conc.added} \times 100$$

Table 5: Accuracy Data for Aspirin by Absorption Correction Method

% Spiking	Total amount of Aspirin (µg/mL)	Amount of Aspirin found (µg/mL) Mean ± S.D (n = 3)	%Recovery	Mean % Recovery
80	10.8	10.75688± 0.065	99.60071	
100	12	12.12498± 0.025	101.0415	100.0905
120	13.2	13.15106± 0.065	99.62926	

Table 6: Accuracy Data for Ticagrelor by Absorption Correction Method

% Spiking	Total amount of Ticagrelor (µg/mL)	Amount of AML found (µg/mL) Mean ± S.D (n = 3)	%Recovery	Mean % Recovery
80	36	36.0732 ± 0.098	100.2032	
100	40	39.2935 ± 0.098	98.23376	99.7870
120	44	44.4065 ± 0.129	100.9239	

Robustness

The assessment of robustness must be taken into account during the development stage and is contingent upon the specific conditions of the procedure. The assay value of the test preparation solution remained unaffected and aligned with that of the actual solution (Table 7-8).

Table 7: Robustness Data Aspirin and Ticagrelor by Absorption Correction Method

Concentration (µg/mL)		% RSD	
Aspirin	**Ticagrelor**	**Aspirin**	**Ticagrelor**
6	20	0.408	0.425
6	12	0.542	0.568
12	6	0.408	0.535

Table 8: Robustness Data Aspirin and Ticagrelor by Absorption Correction Method

Concentration (µg/mL)		% RSD	
Aspirin	**Ticagrelor**	**Aspirin**	**Ticagrelor**
6	20	0.703	0.423
9	30	0.717	0.712
12	40	0.541	0.639

Estimation of Aspirin and Ticagrelor from Their Combination Drug Product by RP-HPLC Method

RP-HPLC method was successfully developed for simultaneous determination of Aspirin and Ticagrelor in tablet dosage form. The results obtained are discussed below.

System suitability

The system suitability parameter was conducted to validate the functionality of the analytical system and its ability to produce accurate and precise results. Six replicates of the reference standard, containing a concentration of 10µg/ml of both aspirin and ticagrelor, were individually injected and a chromatogram was recorded. The outcome of the system suitability parameter is documented in table 9.

Table 9: Results of system suitability parameters

Parameters	Aspirin	Ticagrelor
No. of Theoretical Plates	3253.333	3232.833
Tailing Factor	1.12	1.163
Retention time	4.252±0.001	8.104±0.001344

Linearity and range

The linearity of the analytical method was assessed to determine its capacity to produce test results that exhibit a proportional relationship to the concentration of the analyte in the sample, within a specified range. Various concentrations of standard solutions were prepared and introduced into the High Performance Liquid Chromatography (HPLC) system, and the resulting chromatogram was recorded. The outcomes of linearity are documented in Table 10-11 and Fig 5-6.

Table 10: Response ratio data for linearity of Aspirin

Replicates	Concentration (µg/ml)	Mean AUC	Response Ratio
Rep-1	5	519.427	103.8854
Rep-2	10	1023.192	102.3192
Rep-3	15	1532.948	102.1965
Rep-4	20	2017.082	100.8541
Rep-5	25	2533.370	101.3348
SD 1.037			
%RSD 1.015			

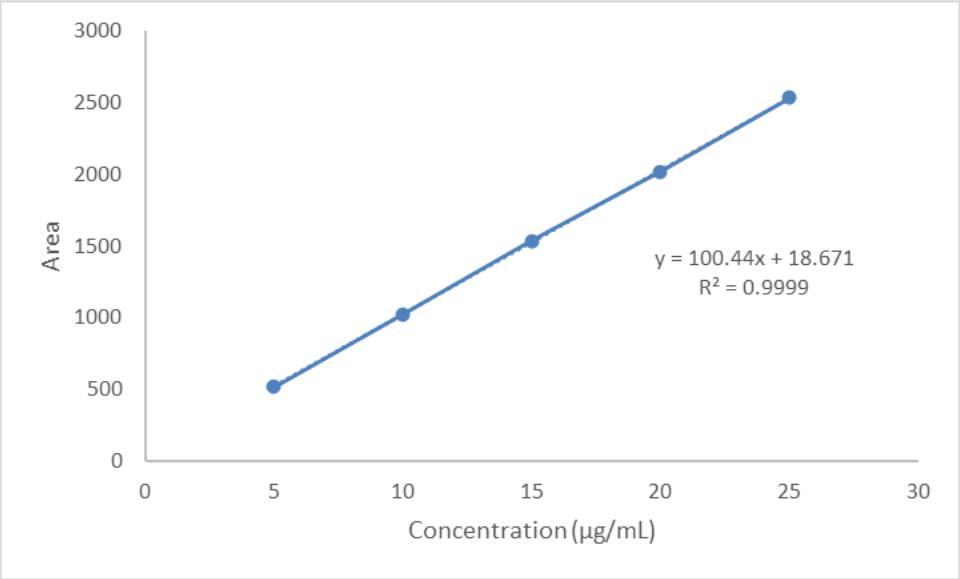

Figure. 5 Calibration Curve for Aspirin by RP-HPLC Method

Table 11: Response ration data for linearity of Ticagrelor

Replicates	Concentration (µg/ml)	Mean AUC	Response Ratio
Rep-1	5	619.035	123.807
Rep-2	10	1246.274	124.6274
Rep-3	15	1865.908	124.3939
Rep-4	20	2445.883	122.2942
Rep-5	25	3028.579	121.1432
SD	1.331		
% RSD	1.080		

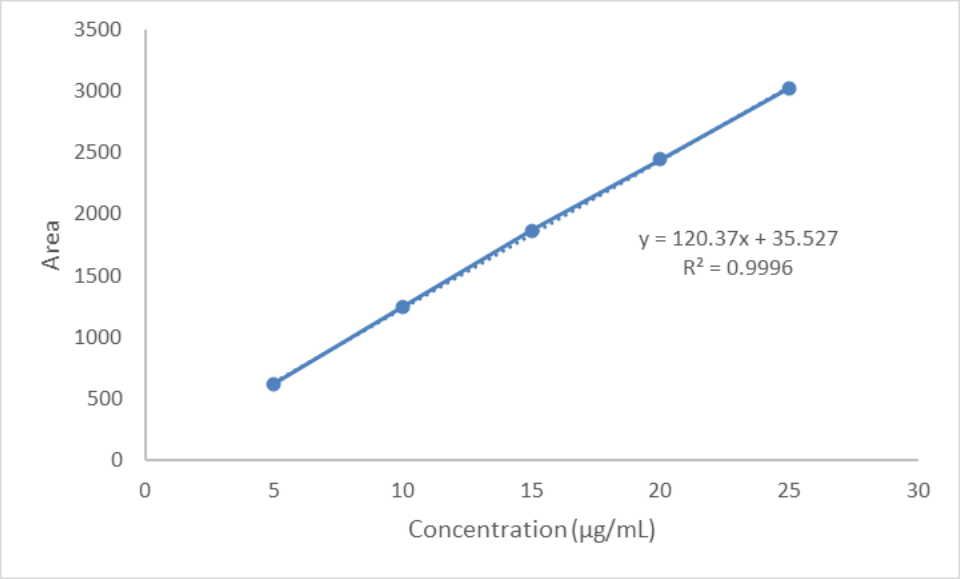

Figure 6: Calibration Curve for Ticagrelor by RP-HPLC Method

Table 12: Results of linearity of Aspirin and Ticagrelor

Parameter	Aspirin	Ticagrelor
Conentration (µg/ml)	5-25	5-25
Correlation Coefficient (r^2)	0.9999	0.9996
Slope (m)	100.44	120.37
Intercept (c)	18.671	35.527

Specificity

Specificity in analytical chemistry is a critical attribute of a method that assesses its ability to unequivocally identify and quantify the analyte(s) of interest while remaining insensitive to potential interference from other components or substances in the sample matrix. In the context of present analysis of Aspirin and Ticagrelor, specificity is a fundamental parameter in ensuring the reliability and accuracy of your results. Specificity of the method was determined and the peaks of diluent, mobile phase and excipients of tablets did not interfere with standard peaks Aspirin and Ticagrelor.

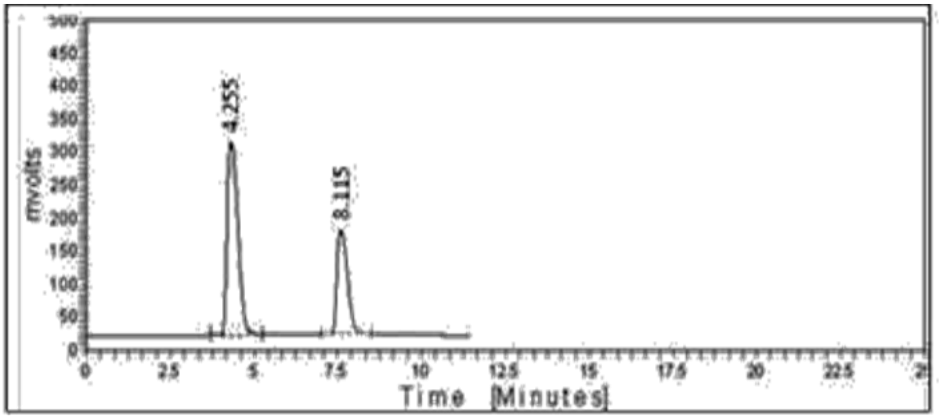

Figure 7: Chromatogram of both the drugs

Accuracy

The assessment of the validity and reliability of the proposed methods was conducted through recovery studies. The recovery of added standards (80%, 100%, and 120%) was determined using three replicates and three levels of concentration. The symbol "%" represents a value that is approximately equal to 100. When the standard deviation (SD) and the relative standard deviation (% RSD) are both less than 2, it indicates the accuracy of the method. The outcome of the recovery study is presented in table 13.

Table 13: Results of recovery study

% Level	% Mean±SD*	
	Aspirin	Ticagrelor
80%	99.10±0.318	99.48±0.215
100%	99.72±0.235	99.14±0.440
120%	99.56±0.254	98.54±0.553

Precision

The precision of the drug was assessed by evaluating its repeatability and intermediate precision. The repeatability result refers to the level of precision achieved when performing a task repeatedly under identical operating conditions within a short time interval. The intermediate precision study quantifies the laboratory variation observed across different days and the analyst-to-analyst variation observed among different analysts. When the values of standard deviation (SD) and relative standard deviation (%RSD) are both less than 2, it indicates that the method has a high level of precision. Result of precision shown in table 14.

Table 14: Results of Precision

Parameter	% Mean±SD*	
	Aspirin	**Ticagrelor**
Repeatability	99.447±0.036	98.790±0.162
Intermediate precision		
Day to day precision	99.276 ±0.041	99.179±0.085
Analyst to Analyst	98.833±1.242	99.125±0.215
Reproducibility	99.172±0.026	

Robustness

The robustness of developed method was checked by changing in the deliberate variationin solvent. Result of robustness shown in table 15-16.

Table 15: Robustness Data for Aspirin and Ticagrelor using HPLC

Concentration (µg/mL)		% RSD	
Aspirin	**Ticagrelor**	**Aspirin**	**Ticagrelor**
80	250	0.4554	0.6759
80	160	0.5734	0.7428
160	80	0.3652	0.4725

Table 16: Robustness Data for Aspirin and Ticagrelor using HPLC

Concentration (µg/mL)		% RSD	
Aspirin	**Ticagrelor**	**Aspirin**	**Ticagrelor**
60	187.5	0.8061	0.5305
80	250	0.5634	0.7352
100	312.5	0.6526	0.8532

LOD and LOQ

The method under consideration demonstrated detection limits and quantitation limits of 0.570µg/ml, 0.350µg/ml, and 0.50µg/ml, respectively. Additionally, the quantitation limits were found to be 1.54µg/ml, 0.95µg/ml, and 1.58µg/ml, respectively, based on the standard deviation of the response and slope. These values satisfy the criteria set for the new method.

The data indicates that the developed analytical method for Aspirin and Ticagrelor analysis is highly precise, with minimal variability in results, particularly during repeatability and intermediate precision tests. The method's robustness and reproducibility are also noteworthy, making it well-suited for the accurate and consistent quantification of these pharmaceutical compounds in various dosage forms.

4. Summary and Conclusion

In summary, the proposed analytical methods for the estimation of Aspirin and Ticagrelor in marketed formulations have successfully achieved the objectives of this research work. These methods stand out for their simplicity, rapidity, accuracy, and reproducibility, making them highly suitable for practical applications in pharmaceutical analysis. The linear range of 5-25 µg/ml for both Aspirin and Ticagrelor, with correlation coefficients close to one, demonstrates the robustness and reliability of the proposed methods.

The validation and reliability of these methods were rigorously assessed through recovery studies, which yielded satisfactory results, ranging from 99.102% to 99.56% for Aspirin and 99.48% to 98.54% for Ticagrelor at various added standard concentrations (80%, 100%, 120%). This indicates that the methods are capable of providing accurate and precise results in the analysis of these pharmaceutical compounds.

Furthermore, precision was evaluated through various parameters, including repeatability, intermediate precision, and reproducibility, all of which demonstrated the consistent and reliable performance of the methods. The robustness of the developed method, tested by introducing deliberate variations in solvent, further confirmed the versatility and adaptability of the methods.

In conclusion, the developed analytical methods offer a cost-effective, rapid, and straightforward solution for the accurate quantification of Aspirin and Ticagrelor. Their precision, low variability and robustness make them well-suited for routine analysis in quality control laboratories, ensuring the production of high-quality pharmaceutical formulations. These methods not only meet the research objectives but also offer practical and reliable tools for pharmaceutical analysis.

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