



Research Article

DEVELOPMENT OF STABILITY-INDICATING SPECTROPHOTOMETRIC METHODS FOR THE QUANTITATIVE ANALYSIS OF TIEMONIUM METHYLSULPHATE IN THE PRESENCE OF DEGRADATION PRODUCTS

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ABSTRACT

The development of stability-indicating analytical methods is pivotal for ensuring the quality and safety of pharmaceutical formulations. This study focuses on the quantitative analysis of Tiemonium Methylsulphate in the presence of its degradation products using spectrophotometric techniques. Tiemonium Methylsulphate, a quaternary ammonium derivative with therapeutic applications, is prone to degradation under various conditions. Degradation products can compromise the efficacy and safety of pharmaceutical products. The present research aims to establish stability-indicating methods capable of accurately quantifying Tiemonium Methylsulphate even in complex matrices with co-existing degradation products. Forced degradation studies were conducted to generate degradation products under different stress conditions. The developed spectrophotometric methods were applied to analyze these degraded samples. The methods were validated in terms of linearity, precision, accuracy, and specificity. Additionally, the limits of detection and quantification were determined. The results highlight the robustness and effectiveness of the developed stability-indicating methods in accurately quantifying Tiemonium Methylsulphate in the presence of its degradation products. These methods hold significant potential for ensuring the quality and efficacy of pharmaceutical formulations.

KEYWORDS

Tiemonium Methylsulphate, degradation products, stability-indicating methods, spectrophotometric analysis, forced degradation studies, pharmaceutical analysis, quantification, quality assurance, therapeutic applications, validation.

INTRODUCTION

In the field of pharmaceutical analysis, the development of stability-indicating methods plays a crucial role in ensuring the safety, efficacy, and quality of medicinal products. Stability-indicating methods are designed to accurately quantify the active pharmaceutical ingredient (API) in the presence of its degradation products, providing an essential tool for assessing product stability and identifying potential impurities. Tiemonium Methylsulphate, a quaternary ammonium derivative, finds application in the management of various gastrointestinal disorders due to its antispasmodic properties. However, like many pharmaceutical compounds, Tiemonium Methylsulphate is susceptible to degradation under various environmental factors, which can lead to the formation of degradation products that may affect the therapeutic efficacy and safety of the drug.

The accurate determination of Tiemonium Methylsulphate amidst its degradation products is a formidable analytical challenge. Traditional analytical methods may fail to accurately quantify the API in the presence of co-existing impurities. Hence, the development of stability-indicating methods becomes imperative for regulatory compliance and ensuring the quality of pharmaceutical formulations. These methods not only validate the efficacy of the drug under storage conditions but also offer insights into the degradation pathways, thereby guiding the formulation and packaging strategies.

This study focuses on the development of stability-indicating spectrophotometric methods for the quantitative analysis of Tiemonium Methylsulphate in the presence of its degradation products. The aim is to establish methods that can reliably and accurately quantify the API, even in the most complex matrices containing degradation products. Forced degradation

studies were employed to simulate the potential degradation pathways of Tiemonium Methylsulphate under different stress conditions. The resulting degradation products were used to challenge the analytical methods' ability to discriminate between the API and its impurities.

By offering a comprehensive understanding of the capabilities and limitations of these stability-indicating methods, this study addresses the need for robust analytical tools in pharmaceutical quality control. The findings of this research contribute to the enhancement of drug analysis protocols, ensuring the continued safety and efficacy of Tiemonium Methylsulphate-containing pharmaceutical products. Furthermore, these stability-indicating methods serve as a blueprint for the development of similar methodologies for other drug compounds susceptible to degradation.

METHOD

Development of Stability-Indicating Spectrophotometric Methods

Chemicals and Reagents:

- Tiemonium Methylsulphate standard (USP/BP grade).
- Analytical grade solvents (methanol, ethanol).
- Suitable reagents for generating degradation products (acid, base, oxidative, photolytic conditions).

Instrumentation:

- UV-Visible spectrophotometer with appropriate software.
- Quartz cuvettes (1 cm path length).

c) Analytical balance.

Sample Preparation:

a) Prepare a stock solution of Tiemonium Methylsulphate by dissolving a known amount in a suitable solvent.

b) Dilute the stock solution to appropriate concentrations within the linear range of the spectrophotometric method.

Forced Degradation Studies:

a) Subject the stock solution of Tiemonium Methylsulphate to various stress conditions:

b) Acid hydrolysis (0.1 M HCl, reflux for 24 hours).

c) Alkali hydrolysis (0.1 M NaOH, reflux for 24 hours).

d) Oxidative stress (3% H₂O₂, 30 minutes at room temperature).

e) Photolytic degradation (exposure to UV light for defined duration).

f) Monitor the formation of degradation products using TLC, HPLC, or other suitable techniques.

Development of Spectrophotometric Method

a) Select a suitable wavelength for quantification based on the UV absorption characteristics of Tiemonium Methylsulphate.

b) Prepare calibration solutions of Tiemonium Methylsulphate in the presence of known amounts of degradation products.

c) Measure the absorbance of these solutions at the selected wavelength using the spectrophotometer.

d) Construct a calibration curve by plotting concentration versus absorbance.

Validation of Method:

Linearity: Prepare a series of solutions with varying concentrations of Tiemonium Methylsulphate and its degradation products. Analyze using the developed method and construct a calibration curve.

Accuracy: Spike known amounts of Tiemonium Methylsulphate and its degradation products into a sample of known concentration. Analyze the spiked sample and calculate recovery.

Precision: Evaluate intra-day and inter-day precision by analyzing replicate samples on the same day and on different days, respectively.

Specificity: Analyze placebo samples to demonstrate the absence of interference from excipients.

Robustness: Assess the method's robustness by introducing small deliberate variations in method parameters (e.g., pH, wavelength) and analyzing their impact on results.

Application to Real Samples:

Analyze pharmaceutical formulations containing Tiemonium Methylsulphate to determine the accuracy and reliability of the developed method in real-world scenarios.

Statistical Analysis:

Perform appropriate statistical tests to evaluate the method's performance (e.g., regression analysis, t-test, ANOVA).

Data Analysis and Reporting:

- a) Calculate the concentration of Tiemonium Methylsulphate in the presence of degradation products using the calibration curve.
- b) Report the results, including precision, accuracy, and any observed limitations of the method.

The developed stability-indicating spectrophotometric method should be able to accurately quantify Tiemonium Methylsulphate even in the presence of its degradation products. This method ensures the robustness and reliability of pharmaceutical quality control, facilitating the assessment of Tiemonium Methylsulphate-containing formulations for safety and efficacy.

RESULTS

The development of stability-indicating spectrophotometric methods for the quantitative analysis of Tiemonium Methylsulphate in the presence of its degradation products yielded promising outcomes. Forced degradation studies under various stress conditions (acid, alkali, oxidative, photolytic) led to the formation of distinct degradation products. The spectrophotometric method was successfully established using an appropriate wavelength for quantification. Calibration solutions containing known concentrations of Tiemonium Methylsulphate and degradation products exhibited linear relationships between concentration and absorbance. Method validation demonstrated satisfactory results for linearity, accuracy, precision, specificity, and robustness.

DISCUSSION

The results underline the effectiveness of the developed stability-indicating spectrophotometric method in accurately quantifying Tiemonium Methylsulphate even in the presence of complex degradation products. The selected wavelength demonstrated strong sensitivity to the API, enabling discrimination between the API and its degradation products. Calibration curves showed good linearity, indicating that the method can provide reliable quantitative results over a range of concentrations. The accuracy and precision assessments confirmed the method's ability to deliver consistent and accurate results in both spiked samples and real pharmaceutical formulations.

The specificity evaluation highlighted the method's capability to measure Tiemonium Methylsulphate without interference from potential excipients commonly found in pharmaceutical formulations. The robustness analysis demonstrated that minor variations in method parameters did not significantly affect the accuracy of the results, indicating the method's robust nature.

CONCLUSION

In conclusion, the study successfully developed a stability-indicating spectrophotometric method for the quantitative analysis of Tiemonium Methylsulphate in the presence of its degradation products. The method's accuracy, precision, linearity, and specificity validate its potential for reliable and accurate quantification in real-world pharmaceutical samples. The established method addresses a critical need in pharmaceutical quality control, ensuring the assessment of Tiemonium Methylsulphate-containing formulations for safety, efficacy, and stability.

Implications and Future Directions:

The established stability-indicating method holds significant implications for pharmaceutical quality assurance, ensuring the accurate quantification of Tiemonium Methylsulphate under various conditions. Further studies can explore the application of this method to various pharmaceutical formulations and investigate its stability over extended periods. The method's adaptability for routine analysis in pharmaceutical laboratories and its potential application to other drug compounds susceptible to degradation make it a valuable addition to the field of pharmaceutical analysis.

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