



Original article

Development and validation of stability indicating RP-HPLC method for simultaneous estimation of silodosin and mirabegron in synthetic mixture

Développement et validation de la méthode RP-HPLC indiquant la stabilité pour l'estimation simultanée de la silodosin et du mirabegron dans un mélange synthétique

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Highlights

- A new stability indicating RP-HPLC method has been developed for simultaneous estimation of silodosin and mirabegron in synthetic mixture.

- The developed method is validated as per ICH guidelines Q1A (R2) including specificity, linearity, precision, accuracy and robustness parameters.
- Carry out forced degradation studies on silodosin and mirabegron drug under acidic, basic, oxidative, photolytic and thermal stress conditions.

Summary

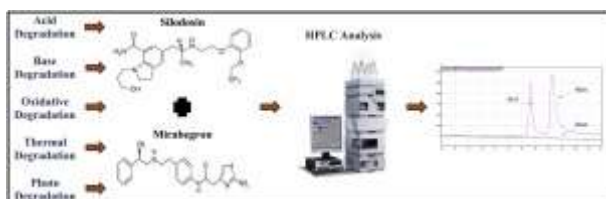
The study focuses to validate and develop a precise, simple and accurate stability indicating RP-HPLC method for estimation simultaneously of and silodosin and mirabegron in synthetic mixture. The chromatographic separation was achieved by using Shimpack Solar C₁₈ column (250×4.5 mm, 5 μm) with acetonitrile: 5 mM ammonium acetate in ratio of 90:10% v/v as a mobile phase at a constant flow rate of about 1.2 mL/min. The development and validation were carried out at detection wavelength of 229 nm. We developed a robust RP-HPLC method, validated for linearity, precision, accuracy, specificity, and system suitability. The method demonstrated excellent linearity with correlation coefficient value r^2 was nearly 0.998 with linearity range 8–18 μg/mL for Silodosin and 24–54 μg/mL for mirabegron. LOD and LOQ were found to be lower; hence, the method is sensitive. Percentage recovery was obtained 99.97% and 99.99% for silodosin and mirabegron, respectively. In case of precision, robustness and repeatability, RSD was found to be less than 2. The validated and developed RP-HPLC method offers an efficient and practical approach for the simultaneous quantification of silodosin and mirabegron in pharmaceutical formulations, making it a valuable tool for quality control and pharmaceutical research.

Résumé

L'objectif de cette étude est de développer et valider et à une méthode indicatrice de stabilité RP-HPLC pour l'estimation simultanée de la silodosin et du mirabegron dans un mélange synthétique. La séparation chromatographique a été réalisée en utilisant une colonne *Shimpack Solar C18* (250 mm Å~ 4,5 mm, 5 μm) avec de l'acétonitrile: 5 mM d'acétate d'ammonium dans un rapport de 90:10% v/v comme phase mobile à un débit constant de 1,2 mL/min. Le développement et la validation ont été réalisés à une longueur d'onde de détection de 229 nm. Cette méthode RP-HPLC robuste a été validée en termes de linéarité, précision, exactitude et spécificité. La méthode a démontré une excellente linéarité avec une valeur de coefficient de corrélation r^2 proche de 0,998 avec une plage de linéarité de 8–

18 µg/mL pour la silodosin et de 24–54 µg/mL pour le mirabegron. La LOD et la LOQ se sont révélées inférieures, la méthode est donc sensible. Le pourcentage de récupération a été obtenu respectivement de 99,97% et 99,99% pour la silodosin et le mirabegron. En termes de précision, de robustesse et de répétabilité, le RSD s'est avéré inférieur à 2. La méthode RP-HPLC validée et développée offre une approche efficace et pratique pour la quantification simultanée de la silodosin et du mirabegron dans les formulations pharmaceutiques, ce qui en fait un outil précieux pour le contrôle qualité et la recherche pharmaceutique.

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Section snippets

Abbreviations

mL

MilliliterIntroduction...

M

Molar...

mg

Milligram...

nm

Nanometer...

cm

Centimeter...

mm

Millimeter...

 μm

Micrometer...

gm

Gram...

min

Minute...

%

Percentage...

 μL

Microliter...

 $\mu\text{g/mL}$

Microgram per milliliter...

mV

Millivolt...

sec

Second...

 r^2

Correlation coefficient...

g/mol

Gram per mole...

mL/min

Milliliter per minute...

 $^{\circ}\text{C}$

Degree Celsius...

hr

Hour...

pKa

Dissociation constant...

N

Normality...

RP

Reverse Phase...

RPC

Reverse Phase Chromatography...

NP

Normal Phase...

S

Slope...

%RSD

Percent Relative Standard Deviation...

Rt

Retention time...

SD

Standard Deviation...

MP

Mobile Phase...

NaOH

Sodium hydroxide...

ACN

Acetonitrile...

API

...

Chemical and reagents

Silodosin and mirabegron were obtained as a gift sample from MSN Laboratory, Hyderabad, India. Methanol, acetonitrile, HPLC grade water and ammonium acetate buffer was purchased from Rankem Chemicals Ltd, Ahmedabad....

Chromatographic conditions

A Shimadzu Co. Kyoto Japan HPLC system equipped with high vacuum pump, a manual injector and diode array detector was used for validation and development purpose. Shippack Solar C₁₈ column (250×4.5 mm, internal diameter, 5 μm particle size) was used to carried out chromatographic...

Establishment of the optimum conditions of the method

While developing the chromatographic method, various parameters like injection volume, flow rate, wavelength, pH of mobile phase, column oven temperature, run time and mobile phase composition are optimized for developing sensitive, accurate, precise and reproducible method which is used for analysis purpose in pharmaceutical industry or laboratories in quality control department....

Column selection

Based on the literature review, C₁₈ reversed-phase column was chosen for initial trial as it is able to resolve a...

LOD & LOQ

Limit of detection and limit of quantitation is calculated based on calibration curve data shown in Table 11....

Conclusion

A stability-indicating RP-HPLC (Reverse phase High-performance liquid chromatography) method was effectively developed and validated for the concurrent quantification of

mirabegron and silodosin in a synthetic mixture. This involved the utilization of a Shimpack Solar C₁₈ column (250×4.5 mm, 5 μm) for separating the two compounds. The mobile phase consisted of acetonitrile and a 5 mM ammonium acetate buffer (pH 6.6) in a ratio of 90:10%v/v. the separation process was conducted under low-pressure...

Human and animal rights

The authors declare that the work described has not involved experimentation on humans or animals....

Informed consent and patient details

The authors declare that this report does not contain any personal information that could lead to the identification of the patient(s) and/or volunteers....

Disclosure of interest

The authors declare that they have no competing interest....

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Author contributions

All authors attest that they meet the current *International Committee of Medical Journal Editors* (ICMJE) criteria for Authorship....

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2024, Analytical Chemistry Letters

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